PLUGGING MATERIALS and the Cementing of Wells

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ТАМПОНАЖНЫЕ МАТЕРИАЛЫ И ТЕХНОЛОГИЯ ЦЕМЕНТИРОВАНИЯ СКВАЖИН

Издание третье, переработанное и дополненное

Издательство «Недра»,
Москва

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PLUGGING MATERIALS and the Cementing of Wells

Translated from the Russian by
S. KITTELL

Mir Publishers · Moscow

First published 1976
Second edition 1985
Revised from the 1982 3rd Russian edition

The Greek Alphabet

Αα	Alpha	Ιι	Iota	Ρρ	Rho
Ββ	Beta	Κϰ	Kappa	Σσ	Sigma
Γγ	Gamma	Λλ	Lambda	Ττ	Tau
Δδ	Delta	Μμ	Mu	Υυ	Upsilon
Εε	Epsilon	Νν	Nu	Φφ	Phi
Ζζ	Zeta	Ξξ	Xi	Χχ	Chi
Ηη	Eta	Οο	Omicron	Ψψ	Psi
Θθϑ	Theta	Ππ	Pi	Ωω	Omega

The Russian Alphabet and Transliteration

Аа	a	Кк	k	Хх	kh
Бб	b	Лл	l	Цц	ts
Вв	v	Мм	m	Чч	ch
Гг	g	Нн	n	Шш	sh
Дд	d	Оо	o	Щщ	shch
Ее	e,	Пп	p	Ъъ	“
Ёё	e, yo	Рр	r	Ыы	y
Жж	zh	Сс	s	Ьь	‘
Зз	z	Тт	t	Ээ	e
Ии	i	Уу	u	Юю	yu
Йй	i, y	Фф	f	Яя	ya

На английском языке

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Preface

This textbook, translated from the third Russian edition, is intended for students at technical schools specializing in the drilling of oil and gas wells. The book outlines the techniques of cementing oil and gas wells, based on current scientific developments and experience gained in applying advanced methods by the Soviet oil-industry specialists, and offers a description of cementing outfit, plugging cements, and chemicals used for their treatment. It also surveys the properties of plugging mixtures (slurries) and cement stone under a variety of geological and technical conditions.

One of the merits of the text is that it describes the composition of plugging cements and techniques employed in their preparation, which is of great importance in training technical personnel at oil fields.

The book will be of particular value in countries where oil is being produced with the participation of the Soviet specialists and with use of the Soviet-made equipment.

Introduction

The drilling of oil and gas wells makes it possible to effect communication of an oil- or gas-bearing stratum with the surface. However, to extract the useful minerals in question, it is necessary to provide a long-lived, stable channel connecting the producing horizon with reservoirs set up at some distance from the well head. For this channel to ensure an unobstructed transportation of fluid or gas, the rock strata must be sealed off from one another and the wall of the hole supported (cased off). Only then will the necessary conditions for a long-term operation of the well be provided.

Wells are cased off with metal pipes which are screwed together to form a column (string) and then lowered into the well to the required depth. These pipes and strings are called *casing*. The cased-off well bore retains its circular cross section for the whole period of drilling or operation of the well.

To seal off the rock strata, a cement mixture (slurry) is pumped into the casing string. The slurry forces out the drilling fluid (mud) contained in the string and then is squeezed into the annular space between the well and the casing (casing clearance) until it reaches the design level mark (Fig. 1). The process of transportation (squeezing) of the cement slurry into the annular space is called *well cementing*.

Russian specialists were the first to use cement for the purpose of cementing wells. Thus, as far back as 1859, the Russian mining engineer G. Romanovsky used cement as a means of controlling the well drowning, and in 1905 A. Bogushchevsky proposed a method of cementing wells, for which he was granted a letters patent in 1906, used by Perkins five years later. Russian mining engineers also knew other methods of cementing wells and employed them in their practical work. The drilling foreman S. Voyslov in his report "On the Research of the Muravievsky Spring" at the meeting of the Mining Engineers' Society on October 23, 1903 described the methods of water control in well drilling and stated that he had successfully applied the cementing of the annular space.

The cementing of wells, especially of deep ones, is the most critical stage in the well construction. The cementing jobs are particularly important because, being final, they may nullify whatever progress made previously. Not infrequently

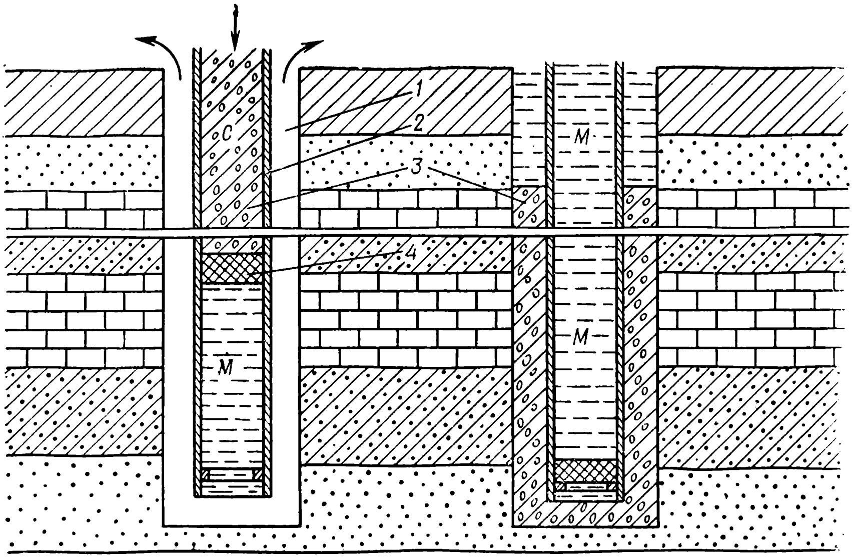


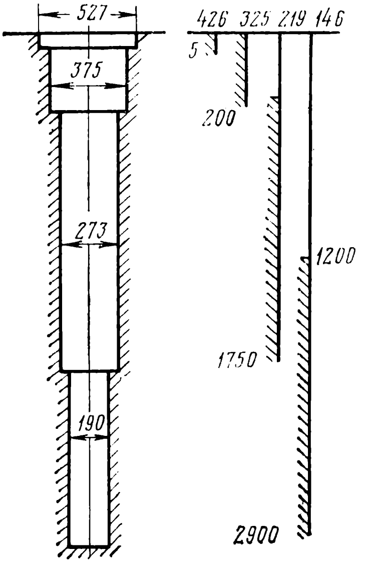
Fig. 1. Illustrating the pumping of the plugging mixture (cement slurry) int the annular space

1 — annular space; *2* — casing string; *3* — cement slurry; *4* — cementing plug; *M* — drilling (clay) mud; *C* — cement slurry (plugging mixture)

an imperfect cementing of wells is the only factor responsible for gas shows, for the formation of gryphons and for the emergence of uncontrolled gassers and gushers causing substantial damage to the national economy. This also results in wrong valuation of the reserves of oil and gas in pay beds; it causes migration of oil and gas into other beds under a lower formation pressure, and contributes to the flooding of producing horizons. At the same time, the reliable sealing off of the rock strata exposed in the course of drilling provides for the conservation of resources which is of great importance both geologically and economically. Therefore, it is quite obvious that the isolation of pay areas, support of wells, sealing off of the rock strata, and quality of well cementing should be given particular attention.

Before drilling a well its casing program is worked out and substantiated. The casing program embraces the arrangement of the casing strings run into the well concentrically, with indication of the string diameters and landing depths, the level mark of the cement slurry squeezed behind the casing, and the diameters of the drill bits used. Figure 2 shows the course of a hole (*a*) and the casing program (*b*). The figures above each casing string indicate the casing diameter, while those at the bottom of the strings, the landing depth. The cementing interval is designated by hatches, the figure above the hachure indicating the level mark of the cement slurry. The gauge numbers of the drill bits used are sometimes also shown on the drawing.

Each casing column, or string, inserted into the well has its own purpose and name. The first and shortest column is called *conductor*. It is set up prior to drilling to protect the ground at the well head from being washed out by the circulating drilling fluid. The second string, which serves to case off insecure uppermost rocks and aquifers, is known as *surface casing*. The lower part of the surface string as well as of all the strings run into the well thereafter terminates in a short thickened pipe commonly referred to as casing shoe. When drilling wells in conditions of permanently frozen ground, both the conductor and the surface casing are designed so as to prevent the thawing-out of the ground. *An intermediate string* (several such strings may be used) is inserted into the well to avert or eliminate troubles which might occur or have already occurred in the course of drilling. The last string, the *production string*, is intended for the exploitation of the producing horizon.



(a) (b)

Fig. 2. Casing program of a well

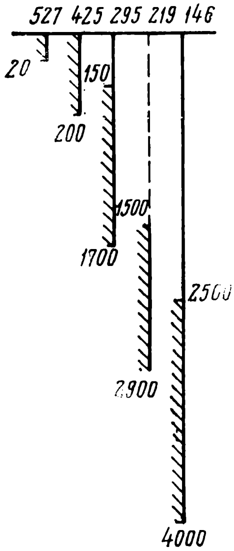


Fig. 3. Casing program of a well with a 219-mm liner

When estimating the number of the casing columns required the conductor and surface casing are not taken account of.

A column which cases off a portion of the well without reaching the head is called *liner*. Liners are frequently used in casing deep wells (Fig. 3).

Oil and gas wells are cemented with a cement mixture (slurry) — a mixture of binding agents (cements) with a definite amount of water (often the mixture contains additions of chemical agents as well). Since mixtures have been developed whose solid phase comprises not only portland cement (if any), it is better to refer to them as plugging mixtures.

The following is a more general definition of plugging mixtures.

A plugging mixture is a material obtained by mixing a plugging cement with water (or some other liquid) treated with some chemicals (if any) to improve the quality of the mixture and the resulting cement stone or to facilitate the casing cementing job.

A plugging (oil well) cement is a material consisting of one or several binders (portland cement, slag, lime, organic materials, etc.) with additions of mineral materials (quartz sand, asbestos, clay, slag, etc.) or organic materials (cotton flocks, cellulose waste, etc.), which, when mixed with water, forms a slurry and then stone of the required properties.

The success of the cementing job largely depends on the equipment and procedures used, as well as on adequate plugging material and preparatory work. The skills of operators and engineering personnel are as important. In proper well cementing all operations should be considered equally significant. Special attention should be paid to the preparation of the well bore, to the choice of case pipes and cement (plugging) mixture (particularly in case of deep hot wells), and to the conduct of the cementing process proper. Long-term operation of the well without recementing is only possible with adequate well casing and reliable sealing off of the rock formations.

Chapter 1

Methods of Casing Cementing

The casing cementing job, regardless of the method, pursues the sole aim of squeezing the drilling fluid out of the annular space through the intermediary of the plugging mixture and lifting the latter to the desired level *. This prevents the possibility for any liquid or gas to migrate from one bed into another through the annular space, ensures permanent isolation of pay areas from extraneous water, supports insecure rocks liable to caving and sloughing, protects the casing string against corrosion by brine water and augments the supporting power of the string.

There are several methods of casing string cementing, which may be divided into two large groups — primary cementing and secondary (remedy) cementing (recementing) methods. Primary cementing is carried out after the drilling has been completed, whereas recementing is usually done after the well has been in operation for some time, in case the leak tightness of the annular space or of the string itself becomes disturbed and the inflow of extraneous water or passage of gas through the cemented annular space, etc., is registered.

Each of the above groups may further be divided into subgroups and subdivisions.

1.1 Primary Cementing Methods

Full Cover Two-Plug Cementing

This is done by means of a special cementing head put on top of the casing string which is inserted into the well, with a cement baffle collar being preliminarily mounted above the casing shoe (Fig. 4). The cementing head (Fig. 5) contains the top cementing plug (also known as follower or wiper plug). Some cementing head designs also provide for the bottom cementing plug to be installed in the head body as well.

The cementing head is connected either to cement pumps or to boring (mud) pumps. After the well has been flushed out,

---

* Remedy cementing (recementing) jobs may be an exception.

the bottom cementing plug is pushed into the casing string (Fig. 4*a*). If the plug is installed in the cementing head, it is first released from the latter and then forced into the string. At the same time the cement pumps and the mixers used to prepare cement slurry (plugging mixture) are put into operation.

After the necessary amount of the cement slurry has been pumped into the well, the top cementing plug is released from the head (Fig. 4*b*). The slurry moves between the two plugs which separate it from the drilling fluid and thus prevent its intermingling with the latter in the casing string (Fig. 4*c*).

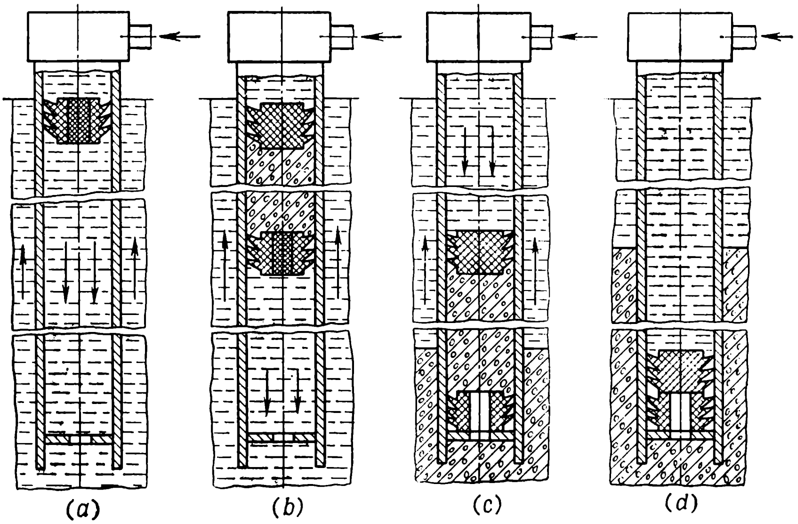


Fig. 4. Illustrating a two-plug cementing job (by stages)

Following the release of the top plug, a displacing fluid (most often drilling mud) is pumped into the well. The fluid forces the cement slurry out of the casing string and squeezes it into the annular space. The squeezing begins the moment the bottom cementing plug is seated on the cement baffle collar and the diaphragm in the plug is broken through (Fig. 4*d*). This is achieved by slightly raising the pressure in the string.

The amount of the displacing fluid to be delivered into the well is calculated beforehand. When there remains 1-2 m^3 of the fluid to be pumped, the pumping intensity is gradually reduced. The operation is continued until the two plugs come together, i. e., the top plug becomes seated on the bottom one. This point is characterized by an increase in the pressure and is referred to as "stop-moment".

Where a spacer fluid is used, the fluid is pumped ahead of and behind the cement slurry. The fluid is intended to prevent the ce-

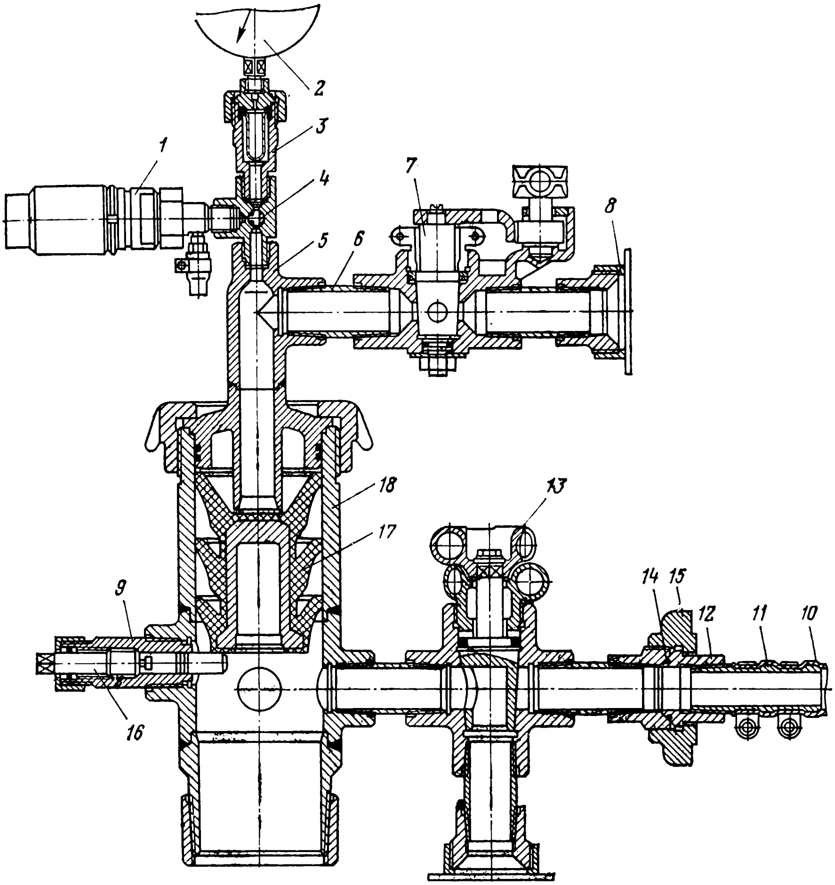


Fig. 5. 2ГУЦ-400 Model cementing head with the top cementing plug installed
1 — safety valve; *2* — pressure gauge; *3* — trap; *4* — tee; *5* — cap; *6* — nipple; *7* — plug valve; *8* — end cap; *9* — sleeve; *10* — hose; *11* — nipple; *12* — quick-disconnect coupling; *13* — three-way plug valve; *14* — seal; *15* — union nut; *16* — stop screw; *17* — cementing plug; *18* — body

ment slurry mingling with the drilling mud and to clean the well bore and walls.

Figure 6 shows various cementing plugs. The plugs are intended to separate the cement slurry from the drilling fluid and to register the end of the cementing process.

Single-Plug Cementing

Oil and gas wells are frequently cemented with only one (the top) cementing plug. This operation is carried out in the same way as two-plug cementing, the only difference being that the bottom plug is absent. The process is considered to be completed when the top plug becomes seated on the cement baffle collar, which is accompanied by an increased pressure in the string.

Cementing of Liners and Lower Sections of Casing Strings

In most cases liners and the lower sections of casing strings (with sectionwise lowering of the strings into the well) are cemented in the same way. Sometimes liners are cemented without cementing plugs. In this case the cementing is done as follows.

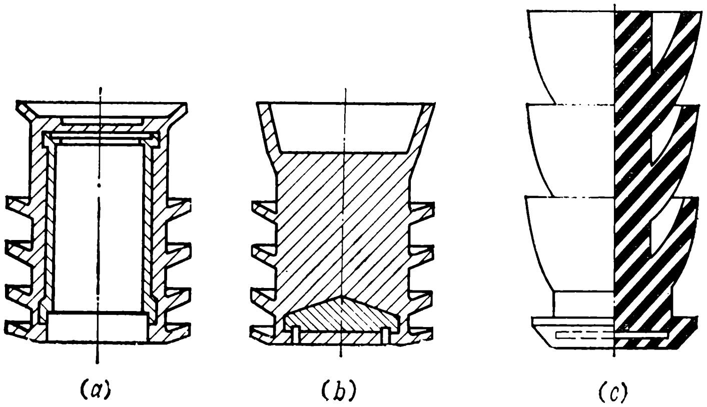


Fig. 6. Cementing plugs
(*a*) bottom plug; (*b*) and (*c*) top (wiper) plugs

After the well bore has been prepared, the liner suspended from the end of the drill string is lowered into the well to the required depth. The liner (casing string section) is joined with the drill pipes by means of a left-hand adapter (a casing substitute with left-hand threads). When lowered into the well, the liner and the drill pipes are filled with drilling mud. After that, the necessary amount of cement slurry is pumped into the pipes and then forced through and out of them by a displacing fluid (drilling mud). The amount of the displacing fluid required is taken to be equal to the inner volume of the drill pipes and liner minus the volume of the cement slurry at the lower portion of the liner (cement plug). Once the slurry is squeezed through, the drill pipes are disconnected from the liner by turning the drill string clockwise and then lifted up several metres. Thereafter the pipes are flushed with drilling mud to clean them and to remove from the well the excessive cement slurry raised above the upper collar of the liner. The flushing is continued until the amount of the drilling mud pumped through the well is 1.5 or 2 times the volume of the well (above the liner). Where a spacer fluid is used, the fluid is pumped ahead of the cement slurry and, sometimes, also behind it.

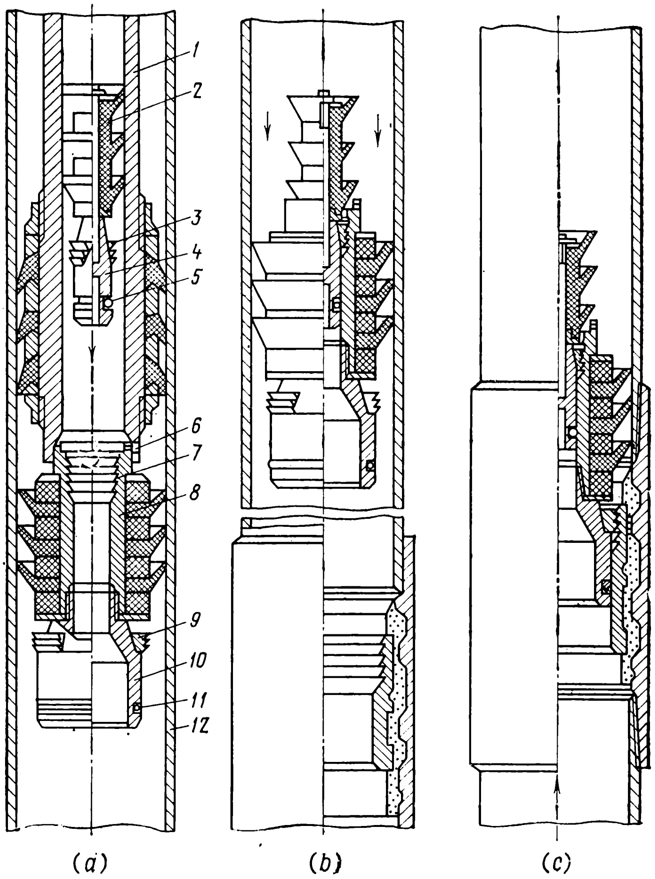


Fig. 7. Illustrating the use of a special cementing plug in cementing a liner
1 and *12* — pipes; *2* and *4* — upper part of cementing plug; *3* and *9* — slips; *5* and *11* — rings; *6* and *7* — connection components; *8* — lower part of cementing plug; *10* — adapter; (*a*), (*b*), and (*c*) plug movement stages

A special cementing plug is successfully used in cementing liners. The plug consists of two parts: the lower part is suspended from shear pins in a special nipple in the liner, while the upper moves through the drill string. When the upper part of the plug becomes seated in a hole provided in the lower part and closes it, an excessive pressure is built up in the string and the pins are sheared off. Both parts of the plug then move together, separating the drilling mud from the cement slurry and making it possible to register the "stop-moment" pressure build-up (Fig. 7). To hold liners and casing-string sections (except for the top one) suspended in the well bore while running them into the well, use is made of special devices, called hangers, of various designs.

After the squeezing of the cement slurry is completed, it is necessary to check the non-return valve for tightness, suspend the liner (casing-string section) from a hanger, disconnect the drill pipes from the casing, flush the well through the drill pipes, and then pull them out. In the course of cementing and also while waiting on cement, i. e., while the rig is shut down to allow cement to set, the liner or casing-string section must remain in tension, with the load on the bottom of the hole, or on the casing-string section that has been previously run into the well, not exceeding 20 to 30 kN.

Full-Hole Cementing (Cementing Through Perforations)

Full-hole cementing is employed in deposits with a low formation pressure or with highly drained beds liable to hydraulic fracturing. In this case use is made of a cement basket which is fitted on the lower portion of the casing string where it is perforated. The cement baffle collar is installed above the perforations. The cementing is carried out as usual, however, the cement slurry here is not squeezed out from under the casing shoe, but through the perforations. The cement basket prevents the slurry from lowering and thus the pressure on the bed below the basket remains unchanged. The well interval above the basket is cemented.

Two-Stage Cementing

Two-stage cementing * is applied when, for geological-engineering reasons, the cement slurry cannot be lifted to the required level at a stretch. The use of this method is advisable in the following instances:

(1) with lost-circulation zones present in underlying beds;

(2) with greatly different temperatures in the zone of lifting of the cement slurry, resulting in a rapid setting of the latter in the lower portion of the well;

(3) with insufficient capacity of the cementing outfit available on the spot.

Two-stage cementing may help cut down cement consumption.

With two-stage cementing, the casing string is cemented in two steps: first the lower portion and then the upper one.

Let us consider in greater detail the two-stage cementing method (Fig. 8). When inserting the casing string, a special cementing collar is mounted on the string at a point corresponding to the selected depth in the well. The collar is provided with holes which are closed while the lower portion of the string is cemented. After the well has been flushed out, the bottom cementing plug is pushed into the string (with single-plug cementing this plug is

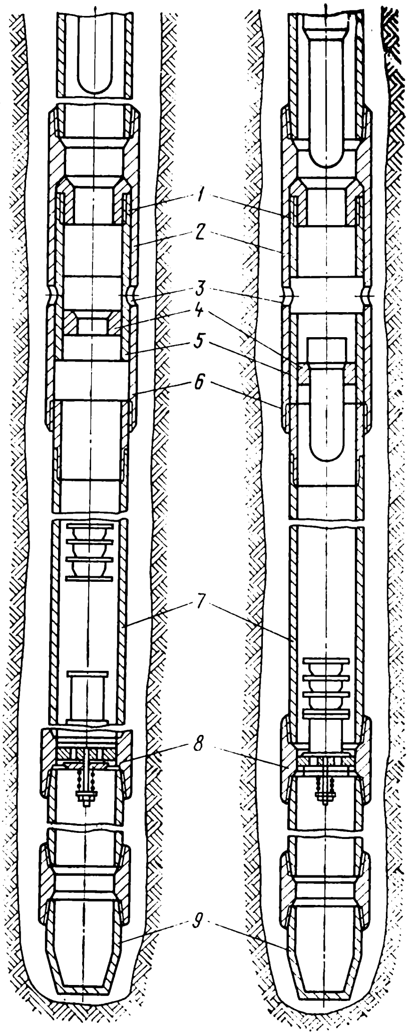
---

* A three-stage cementing method is also used occasionally.

not used). Then the required amount of cement slurry is pumped into the string and after that the top cementing plug is inserted. The slurry is forced into the annular space by displacing fluid taken in an amount approximately equal to the volume of the lower portion of the string. A third plug with a diameter greater than that of the first two plugs is then pushed into the string.

When the top cementing plug is seated on the bottom one, the third plug approaches the cementing collar and shifts a sleeve therein, thus opening the collar holes. The third plug stops in the collar and the displacing fluid now finds its way through the collar holes. After the cement slurry forced above the collar holes is washed out, a fresh portion of slurry is pumped into the string, the slurry flowing through the holes and going up in the annular space above the collar. In the wake of this portion of slurry a fourth plug is dropped down which serves simultaneously as a separating and a closing plug. When the entire cement slurry is squeezed through the collar holes, the fourth plug reaches the collar and, shifting another sleeve, closes the holes. The cementing process is considered to be completed.

The above-described two-stage cementing method is frequently applied with some alterations, use being made of either both or one of the first two cementing plugs.



(a) (b)

Fig. 8. Illustrating two-stage cementing

(a) position before holes in cementing collar become open; (b) position when holes in cementing collar become open; 1 — upper seat; 2 — upper sleeve; 3 — cement slurry exit holes; 4 — lower seat; 5 — lower sleeve; 6 — two-stage cementing collar; 7 — casing string; 8 — cement float valve; 9 — guide shoe

Successful conduct of the cementing process by the two-stage method is dependent primarily on the sound quality of the cemen-

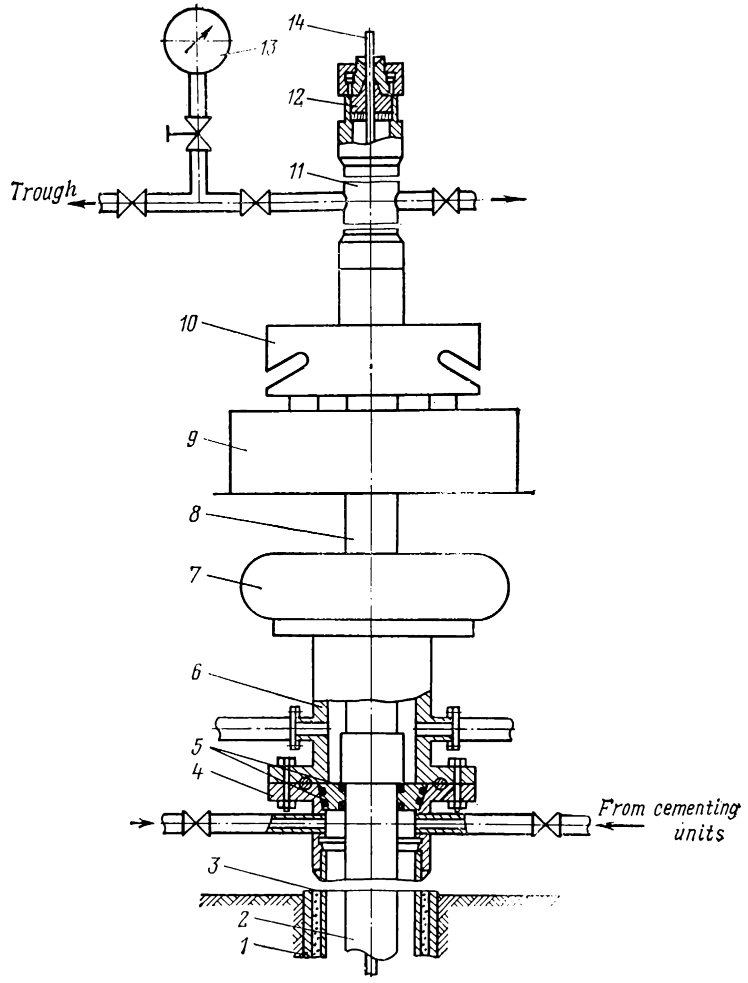


Fig. 10. Wellhead setup for reverse cementing

1 — conductor; *2* — production string; *3* — intermediate string; *4* — casing flange; *5* — seals; *6* — cross; *7* — blowout preventer; *8* — drill pipe 141 mm in diameter; *9* — rotary table; *10* — elevator; *11* — adapter; *12* — sealing gland; *13* — pressure gauge; *14* — logging cable

ments) the moment the cement slurry enters the string, the slurry may be labelled with radioactive isotopes. For this purpose use is made of salts and other materials which change the properties of the slurry and thus make it possible to clearly define the boundary line between the drilling mud and the slurry. The amount of the slurry pumped into the annular space is checked either through calculations or by means of gamma-ray logging equipment (when the slurry is labelled either by using for its preparating collar. Figure *9* illustrates a collar for two-stage cementing. The collar comprises body *2* which is joined with the casing string by means of the upper adapter *3* and the lower adapter *1*. The collar body houses the lower steel cylinder (sleeve) *4* with cast-iron seat *6* for the lower plug, and the upper steel cylinder (sleeve) *5* provided with a cast-iron seat for the upper plug

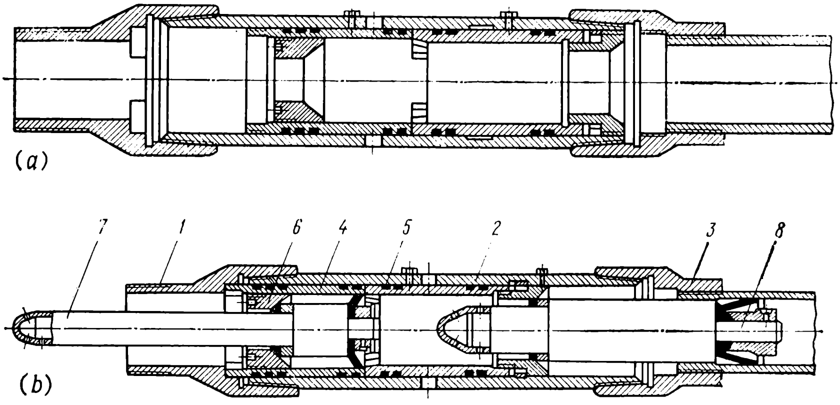


Fig. 9. Two-stage cementing collar
(*a*) position prior to cementing; (*b*) position after cementing

(these plugs are used only at the second stage of cementing). The body is provided with two holes 30 mm in diameter serving to reestablish circulation through the collar, and with four threaded holes to fit stops. The collar set includes two plugs: the lower plug *7* and upper plug *8*.

Reverse Cementing of Wells (Cementing Through the Annular Space)

The reverse cementing is a process whereby the plugging mixture is poured (pumped) into the annular space between the well and the casing (or between different casing columns) and flows down to any required depth either under its own weight or under a pressure produced by pumps.

Though known for a long time, this method has not found wide application because of its inherent disadvantages, the principal one being the difficulty of registering the end of the process, i. e., the moment the mixture enters the shoe of the string being cemented.

Reverse cementing is fairly frequently used as a remedy (recementing) method in cases where production strings develop leaks. It is advisable if the well log includes insecure rock formations susceptible to hydraulic fracturing under low pressures.

Figure 10 shows one of the versions of the wellhead arrangement for reverse cementing. To determine (with special instrution water activated by radioactive isotopes or by means of an ampoule filled with a radioactive isotope, which is dropped through the lubricator into the annular space in the wake of first portion of slurry).

The annular space may be sealed off by using a preventer or special devices. The drilling mud flowing out of the casing is delivered to a trough via special branch pipes.

With reverse cementing, the properties of cement slurry can be adjusted so that when pumped into the annular space the slurry will set almost at once throughout the entire cementing interval.

1.2 Secondary (Remedy) Cementing Methods

Secondary (remedy) cementing or recementing methods are applied in carrying out remedial operations subsequent to detection of leaks, disturbed string sealing or extraneous water entering the well together with oil or gas via string perforations.

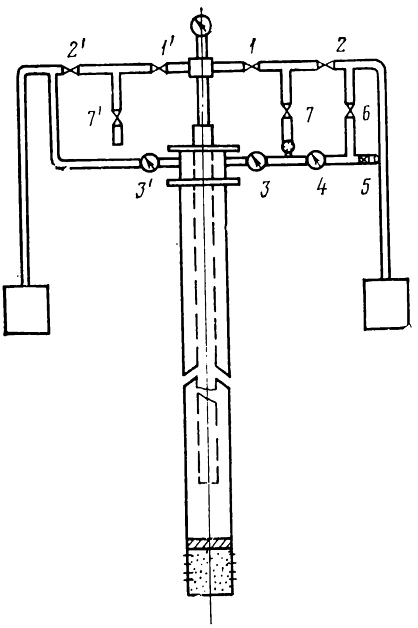


Fig. 11. Wellhead hookup for cementing by the method proposed by N. K. Baybakov

With any recementing method the main operation is the transportation of some amount of cement slurry (determined by calculation) into the well, whether cased off or not.

The slurry is delivered into the well via small-diameter pipes (tubing) and squeezed into the annular space behind them by drilling mud. The pipes are then lifted up and washed through with the mud, and after the slurry had set the cementing process is considered to be completed. The stone thus formed in the well is called cement plug.

In some cases one tries to force the slurry contained in the well into channels in a rock formation or into the holes disturbing the sealing of a string. Then the annular space between the casing pipes or the tubing and the well bore is sealed off and a pressure is built up therein by pumping the drilling mud into the tubing. This operation is known as cementing under pressure or squeeze cementing.

There are several cementing head types and well head and cementing unit (cementer) hookups which help accelerate the setting up of cement plugs in wells. In carrying out isolation jobs, the widest application has found the method proposed by N. Baybakov. With this method two cementing units are employed — one directly for cementing and the other, also serving as a stand-by unit, to speed up the pumping of cement slurry into the well. The cementing units are connected to the well by means of pipes (Fig. 11). The slurry is pumped in via the pipes fitted with valves *1*, *2* and *1'*, *2'*, the valves *3*, *4* and *5* being open. The valves *6*, *7*, *7'* and *3'* are closed. The slurry forces the liquid contained in the well out through the lower pipe provided with valves *3*, *4* and *5*.

To prevent the slurry from rising into the space between the casing string and the tubing inserted in it (sometimes referred to as filling pipes) the valves *4* and *5* are closed the very moment the slurry reaches the shoe of the tubing. It is after this that the squeezing of the slurry into a rock formation through disturbances in the string, or through the screen perforations, begins. The remainder of the slurry is removed from the well through reverse circulation of the drilling mud or water, effected via valves *6*, *4*, *3* and *3'*, the valves *2*, *2'*, and *5* being closed. The liquid flows out of the well through the filling pipes and is discharged via valves *7* and *7'*.

Sometimes cement slurry is transported into the well in a cement dump. The dump comprises a vessel which, prior to its being lowered down the well, is filled with the slurry. It can be made to open after lowering. The slurry then pours out of the dump and settles at the point where a cement plug is to be arranged.

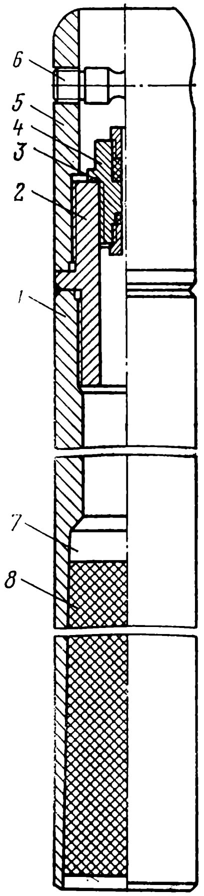


Fig. 12. Shot-plugging tool

Rather labour-consuming work on setting up cement plugs in wells has made it necessary to develop new means for sealing off a portion of the well (casing string). One of them is a shot-plugging tool (SPT) for installing a rubber plug at the desired depth in the casing column. The tool (Fig. 12) consists of body *1* which serves as a barrel and a charge chamber and houses rubber plug *8* and gunpowder charge *7*; adapter *2*; an electric lead-in sealed off by means of ring *4*, two rubber plugs compressed between nipples, and seal *3*; and connecting head *5* with suspension bolt *6*. The charge chamber is separated from the barrel by a disc which is inserted in the tool prior to pressing the plug in the barrel. Once the SPT is lowered into the well to the required depth, the charge is shot and the plug, being forced out of the barrel, expands and closely fits the casing column. Some cement slurry is occasionally poured from a cement dump onto the plug to improve its strength and reliability.

Chapter 2

Technology of Cementing Wells

The technology of cementing oil and gas wells embraces correct implementation of specified operations aimed at supplanting most fully the drilling mud contained in the annular space by a plugging mixture and ensuring sound contact of the latter with the casing and the well walls.

These operations are associated with the movement of slurries — drilling fluids (mostly clay muds) and plugging mixtures — in the casing string and annular space. Therefore one must get familiar with the properties of these slurries to adequately carry out the above-mentioned work.

2.1. Flow Properties of Slurries

Drilling muds and cement slurries are disperse systems whose dispersed phase comprises clay or cement, the dispersion medium being water. The properties of these fluids are closely related to those of the clay or cement and also of the liquid that forms the dispersion medium.

On standing, drilling muds and cement slurries undergo structurization owing to the interaction of the dispersed phase particles (clay, cement) and the dispersion medium molecules, which manifests itself in thickening of the fluids, i. e., in their gelation. As a result, the given fluids possess certain properties which make them somewhat like elastic bodies capable of definite elastic deformation. In cement slurries the structurization of the system increases with time and they gradually harden, losing their fluid properties. In clay muds and cement slurries, clay and cement particles become coated with water films known as the solvate films. Cement particles are wetted well with water and partially dissolve in it. Because the solid disperse-phase particles in clay muds and cement slurries form a three-dimensional structure, these disperse systems, as regards their properties, hold an intermediate position between liquids and solids. Usually, such systems are classed with elastoplastic-viscous bodies. As distinct from water, they remain stationary until the disturbing force applied to them reaches a certain value. For instance, when they are poured into a vessel, the position of their surface in the vessel remains unchanged and they do not flow out when the vessel is slightly inclined, and it is not until the angle of the vessel inclination reaches a certain value that they begin to flow.

As soon as the disturbing force applied to clay muds or cement slurries grows high enough to overcome the strength of their internal structure, they start to deform. However, in contrast to elastic solids, this deformation (plastic) does not disappear (recover) when the disturbing force ceases to act, while the developed stress relaxes.

The science dealing with the study of the deformation and flow of matter is called rheology.

The rheological, or flow, properties of cement slurries are governed by their water-cement ratio, i. e., the relative amount of the mixing water in them, and also by the quantity and quality of the reagents used (if any). If there is more than 50 % (by mass) of water in a slurry, its rheological parameters will be low.

The mechanical properties of elastoplastic-viscous bodies may be characterized by curves that show graphically the dependence of the normal flow velocity gradient du/dn on the shear stress τ (Fig. 13). Such curves are known as flow curves.

Gel strength. The rheological (flow) properties of drilling muds and cement slurries are characterized by their gel strength, viscosity, and yield value. In actual conditions, these characteristics largely depend on the filtration, or water loss, of the muds and slurries, which, in turn, depends on their structurization. The structural strength of a slurry is characterized by the shear stress at which the equilibrium of the slurry is disturbed and it begins to flow. This quantity is called gel strength and is designated by the letter θ. Graphically (Fig. 13) it is represented by the line-segment OA.

The notion of the gel strength θ may be easier understood from the following experiment (Fig. 14). A U-tube of radius R holds a clay mud or cement slurry (with not too high a water loss). The length of the slurry column in the tube is l. The slurry is at rest. Then water is poured into the left-hand limb of the tube. The slurry remains stationary until the water column reaches a definite height, for example, h. As soon as the water pressure on the slurry reaches some critical value the slurry column shifts, rising in the right-hand limb. The slurry rests while it is held back from shifting by its structural strength. This strength may then be calculated. The resting slurry is subjected to the action of force F equal to the weight of the water poured in the left-hand limb of the tube, i. e., $p\pi R^2$ (where p is the pressure of the water column of height h). This force is balanced by the structural strength θ of the slurry effective on the periphery of the tube section of length l, i. e. $\theta 2\pi R l$.

Immediately before the slurry starts moving the entire system is in equilibrium, hence

$$p\pi R^2 = \theta 2\pi R l$$

or

$$\theta = pR/2l \qquad (1)$$

This formula may be used for determining the gel strength of clay and cement slurries.

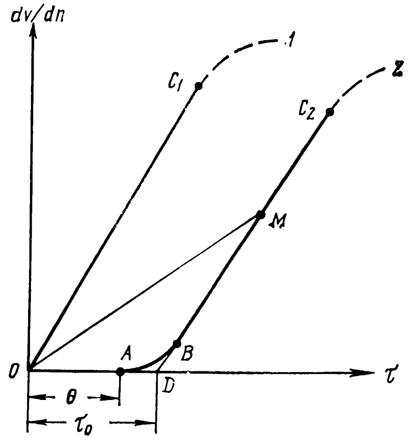


Fig. 13. Flow curves

1 — for vicous fluids; 2 — for viscous-plastic fluids

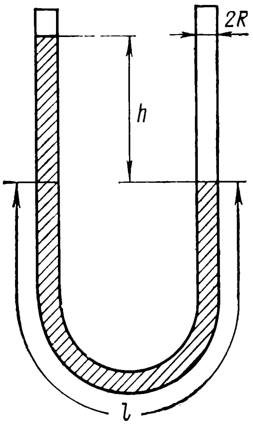


Fig. 14. Schematic diagram of an instrument for determining the gel strength of drilling muds

The gel strength of cement slurries increases with time as a result of physical and chemical processes ocurring in the system. It also increases with rising temperature and pressure, but is reduced when the slurry is stirred with a certain intensity.

The gel strength of clay muds and cement slurries is expressed in pascals (Pa).

Viscosity of slurries. Consider some space filled with a clay mud or cement slurry and confined between two parallel plates A and B (Fig. 15). Assume that both plates move together with the slurry from left to right, as shown by arrows, and that the lower plate B moves with a velocity v while the upper plate A moves faster, with a velocity $v + \Delta v$, due to the application of the force F. Evidently the velocity of individual layers of the slurry rises as they come closer to the plate A. Experiment shows that the force F to be applied to the plate A is directly proportional to the plate area S. This force is equal to the force of resistance offered by the slurry contained between the plates. The layer of the slurry in contact with the plate A adheres to it so that it moves with the same velocity as the plate. The layers more distant from the plate A move slower, and the velocity of the layer in contact with the plate B is equal to that of the plate B. In other words, the velocity of the particles (layers) of the slurry drops from $v + \Delta v$ down to v. Thus, there is a velocity difference between adjacent layers of the slurry (as well as between its individual particles), and the layers slip a little relative to one another, which gives rise to internal friction.

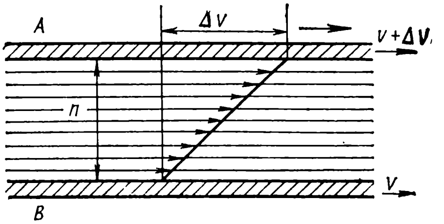


Fig. 15. Illustrating the viscosity of fluids

The viscosity of slurries is a measure of their resistance to the displacement of parallel slurry layers relative to one another. It is designed by the letter μ (mu). Thus,

$$F = \mu S (v/n) \qquad (2)$$

Taking the force per unit area, e. g., per cm², the formula (2) may be written as

$$\tau = F/S = \mu (v/n)$$

or

$$\tau = \mu (dv/dn) \qquad (3)$$

which is known as shear stress formula.

In this formula, dv/dn is the velocity gradient (i. e., the change of velocity dv along section dn), and μ is the coefficient of internal friction or the coefficient of dynamic (absolute) viscosity.

The viscosity μ is the frictional force per unit area at a velocity gradient equal to unity.

In the SI system, the coefficient of dynamic viscosity is measured in newton seconds per square metre (N s/m²).

The formula (3), which is graphically described by the curve *1* of Fig. 13, is valid only for what is known as the Newtonian fluids for which the magnitude of the coefficient of viscosity is independent of the velocity gradient dv/dn. The viscosity of clay muds and cement slurries decreases as dv/dn grows higher. This phenomenon is called viscosity anomaly and is due to the peculiar character of breakage and recovery of bonds between the structure-forming elements upon the flow of the system. While the viscosity of distilled water at 20 °C is 10^{-3} N s/m², that of a freshly prepared cement slurry with a water-cement ratio of 0.5 is $(20 \text{ to } 50) \times 10^{-3}$ N s/m², the flow conditions being the same.

The dual nature of the shear resistance of clay muds and cement slurries (plugging mixtures) allows the resistance to be represented for practical purposes as the sum of two components — a viscous component and a strength component:

$$\tau = \mu\,(dv/dn) + \theta$$

or

$$\tau = \eta\,(dv/dn) + \tau_0 \qquad (4)$$

where η is the structural, or plastic, viscosity of the slurry, which, in contrast to μ, has no strict physical sense, and τ_0 is the yield value of the slurry. Graphically, the rheological equation of clay muds and cement slurries is shown by the curve *2* in Fig. 13.

Yield value. As distinct from the gel strength characterizing the shear stress at the moment the slurry starts flowing (section *AO* in Fig. 13), the yield value expresses the minimum shear stress to be applied to slurry to disturb its state of equilibrium (assuming the slurry to be ideally plastic).

The notion of yield value is conventional. This value cannot be measured directly with any instrument. The yield value (in Pa) is numerically equal to the length of line-segment *OD* (see Fig. 13) on the abscissa axis, cut off by the extension of the rectilinear section BC_2 of curve ABC_2, or it may be found analytically.

In most cases the yield value of clay muds and cement slurries is higher than their gel strength, this being accounted for by the physicochemical nature of these fluids.

In calculations, use is also made of effective viscosity which may be found from formula (4) as follows.

Dividing the left- and right-hand sides of the equation (4) by dv/dn, we get

$$\frac{\tau}{dv/dn} = \frac{\eta\,(dv/dn)}{dv/dn} + \frac{\theta}{dv/dn} = \eta + \frac{\theta}{dv/dn}$$

Denoting $\eta' = \eta + \frac{\theta}{dv/dn}$, we obtain

$$\tau = \eta'\,(dv/dn) \qquad (5)$$

The effective viscosity η' is graphically represented by the slope of a straight line drawn from the origin of coordinates to any point on the flow curve (see, for example, *OM* and AC_2 in Fig. 13). The value of η remains constant throughout the entire length of section BC_2, while η' varies for different points.

2.2. Idea of Slurry Flow

As considered earlier, clay muds and cement slurries start flowing only when the external pressure on a slurry contained, for example, in a tube exceeds certain critical pressure P_{crit}. This pressure is required to overcome the resistance to the flow of slurry, i. e., to overcome the shear stresses active on the tube walls. At the moment the flow commences the whole mass of the slurry is detached from the tube walls and moves as a continuum. A thin fluid layer, called wall boundary layer, remains at the tube walls while the rest of the slurry moves like a solid cylinder or rod. This type of flow is known as *structural* (plug) flow. It is characterized by the presence of a flow core.

As the applied pressure is raised, i. e., as the flow velocity increases, the slurry layers closer to the tube centre move faster, but the very character of the movement of the slurry layers is fairly regular — individual streams do not intermingle and smoothly flow past boundary surfaces. When flowing in a round pipe, concentric cylindrical layers slide relative to one another like telescoping tubes. Such a flow is called *laminar*. However, the notion of laminar flow is not quite applicable to fluids like clay mud and cement slurry. It is more proper to refer to such a flow of slurries as *quasi-laminar* *.

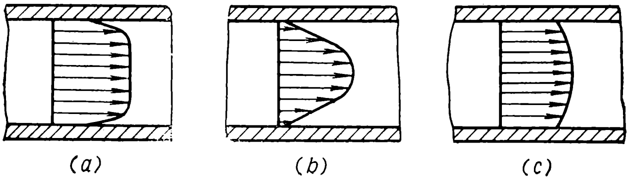


Fig. 16. Duct velocity profiles for different flow conditions

If the flow velocity of the slurry is further enhanced as a result of an increase in the pressure applied, a mixed flow sets in, which then changes to *turbulent* flow. In turbulent flow there is no flow core.

Figures 16*a*, *b* and *c* show the duct velocity profiles for structural, laminar and turbulent flows, respectively.

Turbulent flow of slurries (as well as of all fluids in general) is characterized by a chaotic movement of their individual layers and particles. Since in turbulent flow the number of sliding layers and particles is greater than in laminar and structural flow, the viscosity of the slurries also changes until their structure is completely disrupted.

With transition from structural to turbulent flow, the hydraulic resistances offered by wells, pipes, annular spaces, etc., change. Therefore much attention is paid to the study of conditions under which such a transition occurs. These conditions may be expressed by means of a numerical parameter or criterion which is called Reynolds criterion (or Reynolds number). This criterion is designated Re. Conformably to the study of the flow of fluids such as clay muds and cement slurries, the Reynolds criterion is called generalized Reynolds number and is designated Re′. It is given by the formula

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* Some researchers do not distinguish laminar flow for viscous-plastic fluids at all.

$$\mathrm{Re}' = \frac{vD\rho}{g\eta\left(1 + \frac{\tau_0 D}{6\eta v}\right)} \tag{6}$$

From the formula (6) it is seen that the generalized Reynolds number Re′ depends on the flow velocity v, structural viscosity η, and yield value τ_0, all other things being equal. The other parameters entering into the formula (acceleration due to gravity, g, pipe diameter, D, and density of the flowing slurry, ρ) practically always remain constant throughout the course of cementing.

With increasing flow velocity v and reducing viscosity η and yield value τ_0, the flow of clay muds and cement slurries changes from structural to turbulent. The higher the speed, the less is the time required for the onset of the turbulent flow, provided the flow characteristics of the fluids remain unchanged. With lower numerical values of these characteristics the turbulent flow of the fluids also sets in more rapidly.

2.3. Preparation of Well Bore for Casing and Cementing

Proper preparation of the well bore is of substantial importance both for the correct landing of the casing string and for its adequate cementing.

The well bore is examined to reveal all intervals of the drill string sticking and slacking off, tight-hole and nominal-diameter intervals, and intervals where the angle of deviation and azimuth of the hole alter considerably. After that a program for conditioning and reaming the hole is worked out. Subject to conditioning are all the intervals where the drill string sticking and slacking off have occurred during drilling, and also the tight-hole and nominal-diameter intervals.

If the well is to be cased off with the casing strings of high stiffness, or if the strings are to be run to great depths, the conditioning of the hole involves its reaming with a tool of the so-called rigid make-up. The rigidity of the tool make-up is achieved by fitting out the tool with one or two (rarely three) reamers (drill stabilizers) conformable to the drill bit gauge.

When running the tool of a rigid make-up, especially the one including three-point roller reamers, without limiting the speed of its descent, the rock formations pierced may undergo hydraulic fracturing with subsequent loss of circulation, which complicates subsequent casing cementing job.

There are two methods of limiting the speed of descent of the drill and casing pipes:

(a) with the drilling rigs models Uralmash 4E and Uralmash 6E (Уралмаш 4Э, Уралмаш 6Э) the required braking is effected by operating the boring winch motors as generators, the electric power thus produced being fed back to the supply mains;

(b) with the drilling rig model Uralmash 3D (Уралмаш 3Д) the braking is produced by the winch engines which are motored like compressors with the gear box in reverse ratios.

Table 1 lists the speeds of descent and maximum hook loads for the above drilling rigs.

Table 1

Speeds of Descent and Maximum Hook Loads

Drilling rig Model	Gear engaged	Number of motors used	Maximum hook load, MN	Speed of descent, m/s	Motor speed, rpm
Uralmash 4E	I	2	2	0.26	1035
	II	2	1.73	0.59	1040
Uralmash 6E	I	2	1.2	0.23	778
	II		0.9	0.4	778
Uralmash 3D	I	3	1.7	0.18	—
	II		0.7	0.37	—

Note. 5×6 reeving

After the casing string is lowered home, the drilling mud should be treated once more and the well flushed out at the maximum permissible circulation rate.

Specialists at the All-Union (USSR) Scientific Research Institute for Well Support and Drilling Fluids have proved the advisability of combining the process of preparing the well bore for casing and cementing with that of drilling the well. The preparation of the well bore for casing and cementing directly in the course of drilling allows the process of reaming the well bore with tools of rigid make-up to be completely excluded from the drilling cycle. To achieve this, it is necessary to select the drill bit gauge and the make-up of the bottom part of the drilling tool in accordance with the given permissible deviation intensity of the well bore, the size and rigidity of the casing string run into the well, and the weighted average angle of inclination of the unsupported well bore interval.

The diameter of the drill bit to be used for driling a well that could be cased without resort to reaming should be selected so as to ensure success in running the casing string all the way down to the bottom of the hole. This is done by using the formula

$$D = (3.26 \times 10^4)\frac{m}{EI}(\sin\alpha - \cos\alpha/f) + 0.1745i + d$$

where D is the bit diameter, m, m is the per-meter weight of the casing pipe in the drilling fluid, kg, EI is the rigidity of the casing pipe, N m^2, α is the weighted average angle of inclination of the unsupported well bore interval, degrees, i is the deviation intensity of the well bore in the same interval, degree/10 m, and d is the casing pipe diameter, m.

The rigidity of casing pipes can be found from Table 2.

Table 2

Rigidity of Casing Pipes

Pipe OD, mm	Rigidity, 10^3 N m^2	Pipe OD, mm	Rigidity, 10^3 N m^2
140	1 580	299	20 380
146	1 865	324	27 000
168	3 215	340	30 700
178	3 820	351	34 700
194	5 480	377	44 000
219	7 650	407	57 000
245	10 400	426	64 800
273	15 000	478	93 200

The deviation intensity of the well bore is determined by the formula

$$i = 2a2c \sin\sqrt{\sin^2\Delta\alpha/2 + \sin^2\Delta\varphi/2\,\sin^2\alpha_m}$$

where α_m is the mean angle of inclination of the given well bore interval, degrees, $\Delta\alpha$ is the change in the angle of inclination of the well bore within the limits of the given interval, degrees, and $\Delta\varphi$ is the change in the azimuth angle within the limits of the interval, degrees.

Here, the length of the well bore interval is taken to be the distance between two adjacent points at which inclinometer measurements of the inclination and azimuth of the well bore have been taken.

Examples of Well Bore Diameter Calculation

Example 1. *Given*: The length of the casing interval is 2 000 m; the well bore deviation from the vertical is 600 m. Determine the bit diameter to ensure the passage of a casing string 377 mm in diameter through the well bore all the way down to the bottom.

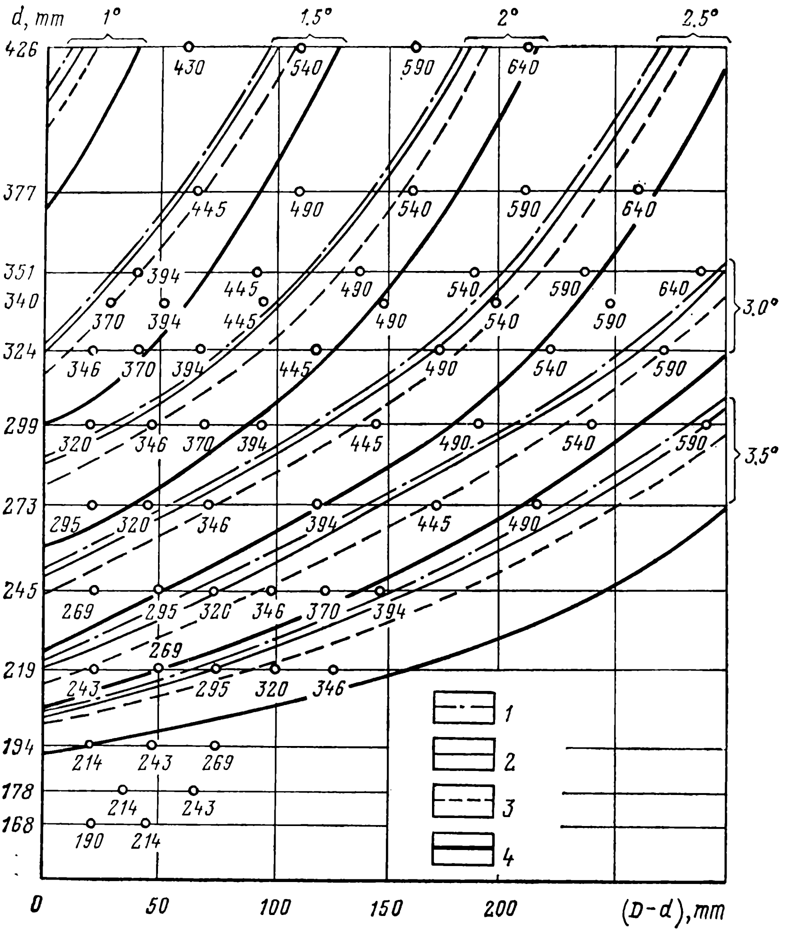


Fig. 17. Nomograph for determining the well bore diameter for casing (with due regard for hole deflection)
1—sin $\alpha_m = 0.05$; 2—sin $\alpha_m = 0.1$; 3—sin $\alpha_m = 0.2$; 4—sin $\alpha_m = 0.4$

Solution. For the given case, we have:

$$EI = 44 \times 10^6 \text{ N m}^2; \quad m = 83.1 \text{ kg/m}; \quad \sin\alpha = 600/2000 = 0.3$$

$$\alpha = 17.5°; \quad \cos\alpha = 0.9537; \quad f = 0.3$$

Hence

$$D = (3.26 \times 10^4)\,\frac{83.1}{44 \times 10^6}\left(0.3 - \frac{0.9537}{0.3}\right) + 0.1745 \times 1.5 + 0.377 = 0.462 \text{ m}$$

Consequently, the well should be drilled with a bit 490 mm in diameter.

If the well bore deviation from the vertical within the limits of the casing interval in the above example is taken to be 300 m ($\sin\alpha = 0.15$), a drill bit 445 mm in diameter will then suffice:

$$D = (3.26 \times 10^4)\,\frac{83.1}{44 \times 10^6}\left(0.15 - \frac{0.9894}{0.3}\right) + 0.1745 \times 1.5 + 0.377 = 0.443 \text{ m.}$$

Table 3

Recommended Minimum Drill Collar Diameters

Casing string OD, mm		Effective well bore diameter, mm	Minimum necessary drill collar diameter, mm, for drill bit diameter, mm														
Casing pipe, OD, mm	Coupling OD, mm		640	590	540	490	445	394	370	346	320	295	269	243	214	190	161
426	451	439	299	299	340	407											
377	402	390		273	273	299	340										
351	376	364			254	254	299	340									
324	351	338				229	229	229									
299	324	312					229	229	254	299							
273	299	286						203	203	229	254	254*					
245	270	261										229	254*				
219	245	235											203	203*			
194	216	207											178	178	178*		
178	198	198												178	203		
168	188	188												146	178	178*	
146	166	166													133	146	146*
140	159	159													133	133	133*

Notes: 1. Drill collars should be at least 1 m long. 2. Asterisked are flush-joint connections.

The necessary drill bit diameter can also be found promptly by using the nomograph of Fig. 17.

Example 2. *Given*: The length of the casing interval is 2 000 m; the well bore deviation from the vertical is 400 m ($\sin\alpha = 0.2$); the mean deviation intensity is 2.5 deg/10 m. Using the nomograph of Fig. 17, find the bit diameter necessary to ensure the passage of a casing string 273 mm in diameter down the well bore.

Solution. From the point corresponding to the casing string diameter on the Y-axis draw a horizontal line to intersect the curve corresponding to $\sin\alpha = 0.2$ in the region of deviation intensity of 2.5 deg/10 m. The point of intersection indicates that the bit diameter should be at least 346 mm.

With the bit diameter determined by the above method, it is necessary to use drill collars in the drilling string above the bit in order to ensure the passage of the casing string through the well bore. Table 3 lists the recommended drill collar diameters. If suitable drill collars are unavailable, use can be made of the above-bit gaugers (calibrators) designed at the All-Union Scientific Research Institute for Well Support and Drilling Fluids.

2.4. Determining Well Bore Configuration and Volume

Finding the cross section of a well bore from the results of caliper logging leads to errors in calculating the required amount of plugging materials for cementing the well, because the ordinary hole caliper is designed to read the diameter of a well bore section as that of a perfect circle. In actual fact, however, a borehole may have various cross-sectional shapes, depending on the geological and technical conditions of drilling and the properties of the rocks traversed by the hole.

To obtain more reliable results in determining the configuration of a well bore, use is made of a section gauge that is capable of recording three curves in a single run, two of the curves (section gauge log) characterizing changes in the well bore diameter in two mutually perpendicular planes and the third curve (caliper log) registering the averaged bore diameter.

To aid in interpreting section gauge logs, Fig. 18 shows several versions of such logs that can be met with in practice and indicates the corresponding mean bore diameters.

When both curves of the section gauge log coincide with the gauge-hole line (d_{bit}), the cross section of the well bore has the shape of a circumference with a diameter close to that of the drill bit.

If the two curves of the section gauge log coincide and are located to the right of the gauge-line, the bore diameter is greater than the bit diameter (a cave with a round cross section), the distance between the curves and the gauge-hole line increasing with the size of the cave.

When the section gauge log curves are located both to the right of the d_{bit} line but do not coincide, the cross section of the

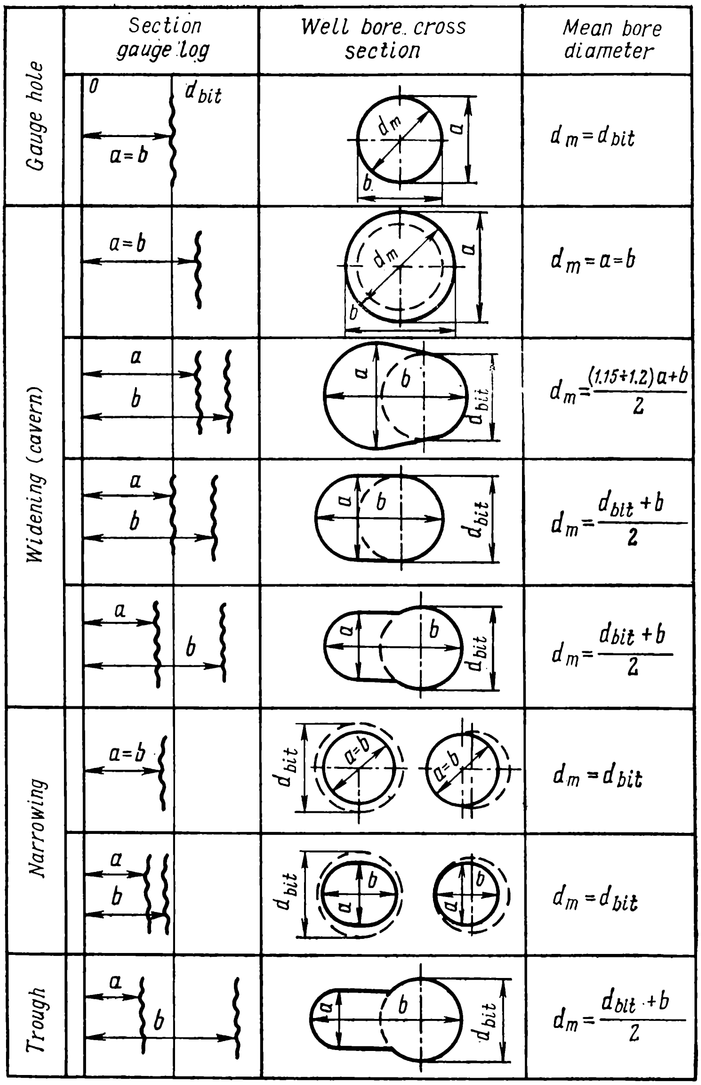


Fig. 18. Well bore cross-sectional shapes according to section gauge logging data

well bore is an oval (an oval cavity), and the more distant are the curves from each other, the more elongated is the cross-sectional shape of this type of cavern.

If both curves of the section gauge log are located to the left of the d_{bit} line and coincide, the cross-sectional shape of the well bore is a circumference with a diameter less than the drill bit diameter; with the curves not coincident, the cross-sectional shape of the well is an oval whose size across the major axis is smaller than the bit diameter.

When the section gauge log curves are located one on each side of the d_{bit} line, the cross section of the well bore is trough-shaped (trough-shaped cuts), and the deeper the trough in the well wall, the more distant are the curves from each other.

After interpretation, the section gauge log is divided into individual sections representing trough-shaped cuts, cavities, narrowings, and gauge-hole intervals. After that, the mean bore diameter is calculated as illustrated in Fig. 19.

Then the volume of each well bore interval is determined and the total volume of the annular space (casing clearance) calculated.

In the case of trough-shaped cuts, the section gauge log is used to determine the quantity *b* that represents, on a certain scale, the maximum cross-sectional size of the well bore. The half-sum of this quantity and the bit diameter gives the mean diameter of the trough-shaped cut interval.

Example. Determine the mean diameter d_m and the volume V of the well bore in the interval from 625 to 715 m, which features a trough-shaped cut (Fig. 19*a*); $b = 615$ mm, $l = 90$ m (the length of the cut), $d_{bit} = 394$ mm (bit diameter).

Solution:

$$d_m = \frac{394 + 615}{2} = 505 \text{ mm};$$

$$V = 0.785 \times 0.505^2 \times 90 = 18 \text{ m}^3.$$

The volume of this well bore interval, found on the basis of caliper logging data, amounted to 10.37 m^3 only.

For cavities of round cross-sectional shape, the quantities *a* and *b* are equal and correspond to the mean well bore diameter in the interval under consideration (Fig. 19*b*).

For cavities with $a < d_{bit}$, the quantity *b* is determined from the section gauge log. The half-sum of this quantity and the drill bit diameter will give the mean diameter of the given well bore section.

Example. Determine d_m and V in the well bore interval from 1870 to 1945 m, which is a cavity of an oval cross-sectional shape (Fig. 19*c*); $a = 320$ mm, $b = 480$ mm, $l = 75$ m, $d_{bit} = 394$ mm.

Solution:

$$d_m = \frac{394 + 480}{2} = 437 \text{ mm};$$

$$V = 0.785 \times 0.437^2 \times 75 = 11.3 \text{ m}^3.$$

The volume of the same well bore interval, determined from caliper logging data, amounted to 9.8 m^3.

For cavities with $a > d_{bit}$, the quantities *a* and *b* are found from the section gauge log. The half-sum of *a* multiplied by 1.15

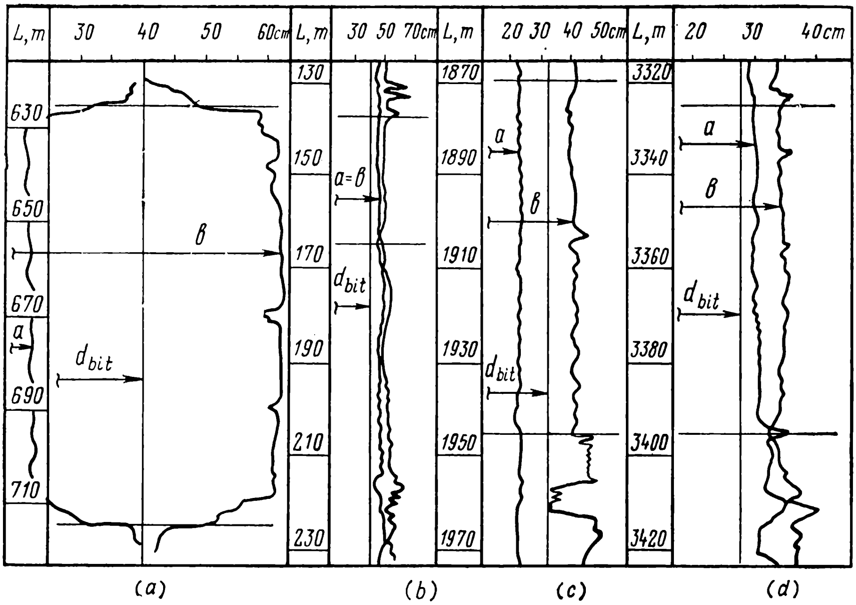


Fig. 19. Section gauge logs

to 1.2 and *b* will be the mean diameter of the given well bore section (Fig. 19*d*).

When making calculations for gauge-hole or narrowed well bore intervals, the mean diameter of a given well bore section is taken to be equal to the drill bit diameter.

2.5. Improving the Quality of Well Cementing

The leak tightness of the casing string and the absence of communication between the rock formations are indicative of a sound quality of the well support.

However, the leak tightness of the string alone does not determine the quality of the well support as a whole, the absence of communication between the rock formations being an indispensable condition for ensuring high quality of the support.

The quality of the well cementing depends on a number of geological and technological factors.

1. Geological factors: the absence or presence of a fluid and its nature (gas, water, oil); dislocations; hydraulic fracturing gradient; lithology of the rock formations (particularly the presence of salts); formation water mineralization; temperature and pressure.
2. Technological factors: casing program; well bore condition; quality of the drilling mud; cementing outfit available; properties

of the plugging mixture used; application of the latest techniques (equipment, spacer fluids, slurry flow conditions, etc.).

Some of the above factors can be taken account of, changed or adjusted in the course of cementing.

The cementing may be considered ideal if the entire drilling mud is forced out by a plugging mixture of good sedimentation stability. Once the mixture has set and hardened, the casing string, equally spaced from the walls of the hole, is surrounded by a stone of the hardened mixture in contact with (or, better, adhered to) the rock formations pierced by the well and with the metal of the string. There will be no channels in the annular space filled by the stone.

2.6. Technological Parameters

The expulsion of the drilling mud by the cement slurry. It is considered an established fact that an increase in the upward annular velocity of the cement slurry makes for better quality of the well cementing, since this provides for a more perfect expulsion of the drilling mud from the annular space.

But the main factor determining the extent of expulsion of the drilling mud out of the well is the nature of the cement slurry flow in the annular space. It is clear that the turbulent flow is most effective.

The upward annular velocity of the cement slurry cannot be practically varied within broad limits, for with too high an upward velocity the hydrodynamic load on the rock formations is greatly increased and this may result in their fracturing. Besides, increasing the velocity within the boundaries of a given type of flow will apparently have no beneficial effect on the quality of the well cementing.

To lower the critical velocity, the viscosity and yield value of the plugging mixture should be reduced by chemical treatment.

The pressures developing in the course of cementing the wells can be greatly reduced, and the amount of the drilling mud expelled, increased, if the cement slurry is treated with special reagents — thinners (viscosity and yield value reducing agents). These include sulphite-cellulose liquor, some high-molecular organic compounds, reagents with calcium lignin sulpho-aluminates as the base, etc.

The use of centralizers. The casing string is centred by means of centralizers to minimize the number of pinched zones with irretrievable drilling mud and, hence, to decrease the amount of the drilling mud expelled. Presented in Fig. 20 are the most widely used spring-type centralizers.

The spacing of the centralizers should be selected analytically.

Reciprocation (rotation) of casing strings and the use of scratchers. The quality of the cementing of wells is greatly improved if the casing string is reciprocated or rotated in the well while the cement slurry rises in the annular space. In this case the drilling mud is most effectively replaced by the cement slurry. The mud moves and its structure becomes disturbed, it is entrained by the casing string moving upwards and is forced towards the well head by the flow of the cement slurry.

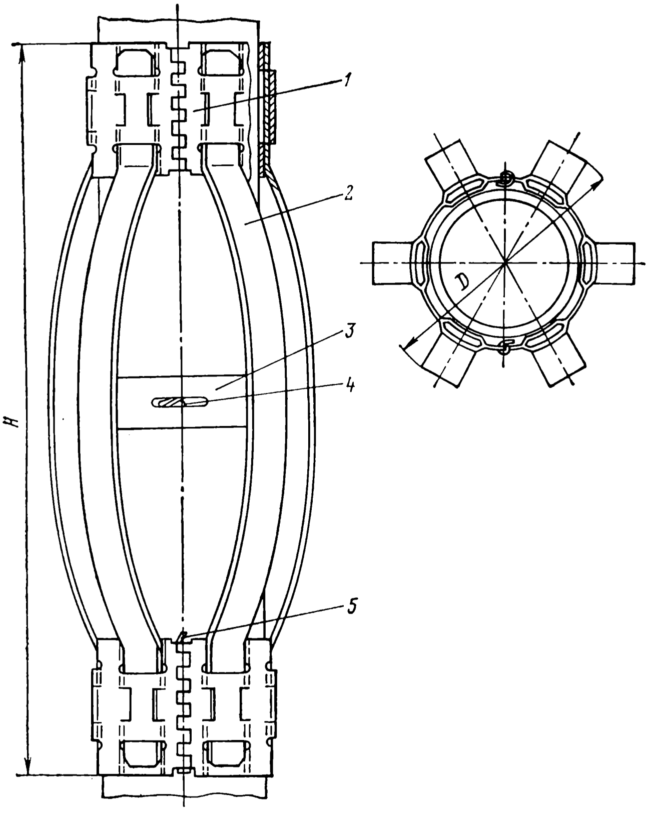


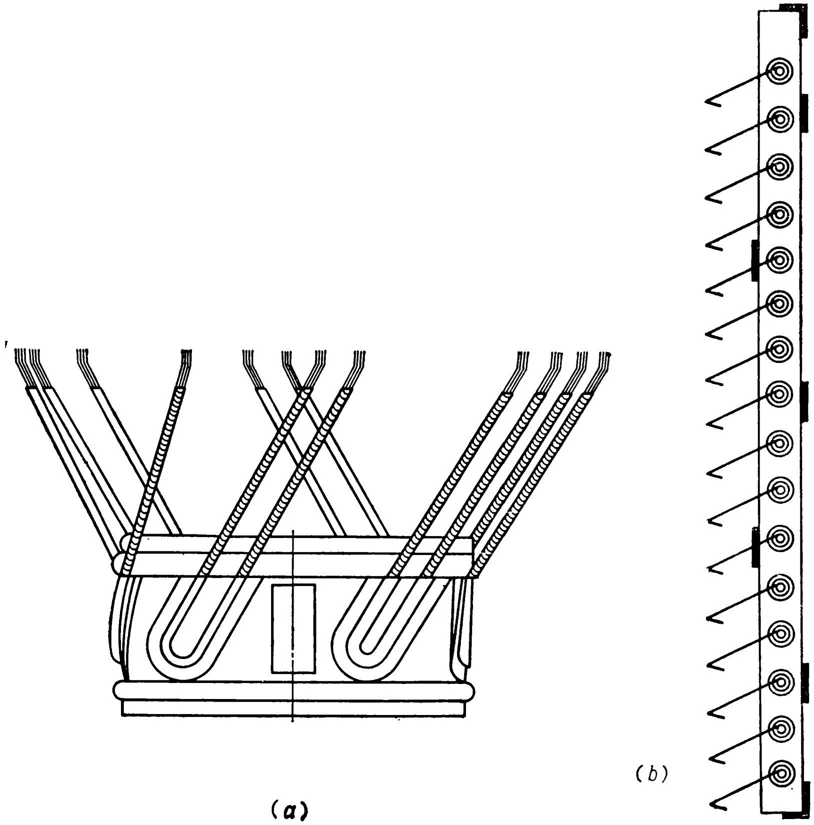
Fig. 20. Centralizer

When cementing casing strings with reciprocation, the probability of their being damaged due to additional stresses arising from the pumping of the slurry and also from temperature changes is greatly reduced.

Substantial improvement in separating the rock formation is also impossible without the removal of the mud cake within the producing intervals. Special literature recommends the following three methods for cleaning the well walls from the mud cake: chemical. hydraulic, and mechanical.

None of the chemical and hydraulic methods for cleaning the well walls from the mud cake has as yet found practical application.

Scratchers for the mechanical removal of the mud cake from the well walls are most widely used in the field. There are two groups of the wall scratchers: radial (Fig. 21*a*) and axial, or comb-type (Fig. 21*b*). The former embrace the casing along its perimeter, whereas the latter are mounted along the generatrix of the casing string.


(*a*)

(*b*)

Fig. 21. Scratchers

(*a*) radial; (*b*) comb-type (axial)

When the string is reciprocated or rotated the scratchers remove the mud cake and the mixture flowing from below carries it to the well head.

2.7. Spacer (Displacement) Fluids

The use of spacer, or displacement, fluids is one of the principal measures to improve the quality of well support. At the present time, there are no universal spacer fluids suitable for any drilling conditions. In current use in the USSR are the following kinds of spacer fluid: water, petroleum derivatives, weighted spacer fluids (saline and polymer-base), hydrochloric acid solutions, compound, aerated, erosion, and non-freezing spacer fluids, spacer fluids of low water loss, and viscoelastic spacer fluids.

Displacement fluids whose viscosity and density exceed those of the fluid being displaced possess better expulsive ability. To diminish the effect of intermingling of fluids on the properties of spacer fluids in the course of their movement in the annular space during cementing and to improve the displacement efficiency, it is necessary that the viscosity and density of the spacer fluids, being higher than those of the fluid being displaced, should be equal to the mean viscosity and density of the fluids in contact with them. On the other hand, to ensure effective removal of drilling mud residues from the well walls and cavities, the spacer fluid must have a good washing ability, i. e., it must have a low viscosity and a minimal density. Improved efficiency can be attained by using compound spacer fluids.

Types of spacer fluids and their application techniques. Water has found application as a spacer fluid in many regions of this country. It washes away well both drilling muds and cement slurries. The use of water as a spacer fluid is recommended in cementing wells drilled in secure rocks not liable to swelling upon a short-term action of a stream of water.

Petroleum and its derivatives are recommended for use as a spacer fluid only where the drilling employs oil emulsion muds or the well bore is cemented with oil emulsion plugging mixtures. Petroleum-base spacer fluids (petroleum, diesel fuel) make for better turbulization conditions in the zone of their intermingling with the drilling mud and improve displacement efficiency.

Weighted spacer fluids are recommended for use in conditions where the application of large quantities of light fluids entails the danger of blowouts or borehole wall collapse and cavings and also where highly cavernous zones are present in the well bore.

Spacer fluids with aqueous solutions of salts ($NaCl$, $CaCl_2$, etc.) as their base have relatively high densities ($\rho = 1$ to 1.6 g/cm^3) compared to water and petroleum derivatives. The use of such fluids is especially effective where the geologic section contains salt domes. The fluids dehydrate mud cakes and reduce their volume and can be used in practically any amounts, so that the drilling mud can be completely replaced by them. Other, heavier spacer fluids are also known.

Acid solutions are intended for removing mud cakes and drilling mud residues from borehole walls in oil-bearing intervals. Such spacer fluids are recommended for use in cementing wells with especially high requirements as to the quality of their sealing. In particular, this applies to wells drilled with carbonate-clay mud flushing in oil fields featuring reservoirs with bottom water or oil-bearing beds separated from aquifers by thin beds of rock.

In use are 8- to 15-percent aqueous solutions of hydrochloric acid or a 20-percent aqueous solution of sulfamic acid.

To break down the mud cake, the volume of the spacer fluid used should be equal to the volume of the annular space in the oil-bearing interval.

Aerated spacer fluids are recommended for use in cases where the well log contains lost circulation zones that make it difficult to cement the well if its imperfection coefficient with regard to bore widening is high.

The use of water-sand erosion spacer fluids is advisable in cementing wells whose walls are made up of clay rocks and have large cavities.

The use of water as a spacer fluid is impermissible in cementing wells drilled in permafrost, since water may freeze in the annular space and crush the casing.

A non-freezing spacer fluid consists of a 30-percent aqueous solution of diethylene glycol and quartz sand 0.1 to 0.15 mm in grain size at a concentration of 40 g/l. The freezing point of such a suspension is around minus 30 °C which is substantially lower than the temperatures prevailing in permafrost and thus makes the cement job possible in winter time.

Spacer fluids of low water loss are employed in casing cementing jobs in deposits with low formation pressure gradients, where the well log contains lost-circulation formations or interlayers of incompetent rocks prone to sloughing and caving upon the action of water, and also when running casing in sections joined in the open hole. The ingress of spacer-fluid water into the pay horizons of oil wells is especially undesirable, since this reduces the rock permeability in their bottom-hole formation zones.

With the existing cementing techniques, viscoelastic spacer fluids provide for complete displacement of the drilling mud by the cement slurry and prevent the intermingling of the slurry and the mud contained in the well. The capability of such a fluid of assuming the shape of the channel it is flowing through ensures complete displacement of the drilling mud from both narrowed and widened sections of the well bore. These fluids, however, possess a number of shortcomings that for the present limit their application.

The amount of a viscoelastic spacer fluid necessary for cementing is calculated so as to have a column 20 to 25 m high in the annular space.

Chapter 3

Cementing Units and Cement Mixers

In the USSR, over 20 thousand oil and gas wells are being cemented annually. This process is carried out by means of a complex of special equipment (cementing outfit) set up in accordance with a preplanned layout. Illustrated in Fig. 22*a* is one of such layouts of cementing equipment, complete with the necessary piping, suitable for a case where 40 to 60 t of a dry plugging material are required to prepare the necessary amount of cement slurry. Figure 22*b* shows a cementing equipment layout using an "averaging" tank (i. e., a tank for "averaging" the plugging mixture parameters).

3.1. Cementing Units

Cementing units, or cementers, are intended for pumping the plugging mixture (cement slurry) and the displacing fluid into the well and also for delivering the mixing fluid (e. g., water) to the mixing unit (cement mixer) when preparing the slurry. In addition, cementing units are employed to wash out or force through sand plugs in wells, to hydraulically test pipes, strings of pipe, and manifolds, to mix hydraulically the slurry, and so on.

Cementers are, as a rule, designed as self-propelled units mounted on motor truck chassis, in which case they are also referred to as cementing trucks; there are cementers of special construction (skid-mounted, tracked, in unit form) suitable for employment under various conditions.

Consider the design of and technical data on some cementing units.

Technical Data on ЦА-320M Model Cementing Unit

Type	Self-propelled, truck-mounted
Overall dimensions, mm:	
length	10 425
width	2 650
height	3 225
Total mass, fully loaded, kg	17 500
Maximum power, kW	177
Cementing pump:	
Model	9T
Type	Horizontal, two-piston, double-acting
Maximum pressure at a delivery of 2.9 l/s, MPa	32
Maximum delivery at a pressure of 4 MPa, l/s	23
Hydraulic power, kW	93
Gear ratio of globoid worm gearing	20.5
Piston stroke, mm	250

Auxiliary engine:	
Model	ГАЗ-51А
Maximum power, kW	52
Maximum speed, rpm	2800
Water pump:	
Model	1B
Type	Vertical, three-plunger, single-acting
Plunger diameter, mm	125
Plunger stroke, mm	170
Crankshaft speed, rpm	140
Delivery, l/s	13
Pressure, MPa	1.5
Gear engaged	II
Capacity, m^3:	
measuring tank	6.4 (two compartments of 3.2-m^3 capacity)
cement tank	1.5
Nominal diameter of intake pipe flow section of 9T and 1B Model pumps, mm	100

The ЦА-320М Model cementing unit (Fig. 23) is mounted on the chassis of the КрАЗ-257 Model motor truck 10 to 12 t in load-carrying capacity, powered by a four-stroke internal combustion engine. All pieces of the cementer equipment — the 9T Model two-piston cementing pump with a protective shroud, the 1B Model vertical three-plunger water pump, the ГАЗ-51А Model water-pump drive engine with a gearbox, the measuring tank, pipelines and fittings, etc. — are mounted on suitable frames held to the truck chassis. The operating conditions of the cementing unit are listed in Table 4.

Table 4

Operating Conditions of the ЦА-320М Model Cementing Unit (calculated for volumetric efficiency $\eta = 0.9$)

Mode of operation	Engine speed, rpm	Gear engaged	Pump speed, rpm	Pressure, MPa, at cylinder sleeve diameter of, mm			Delivery, l/s, at cylinder sleeve diameter of, mm		
				100	115	127	100	115	127
Maximum-delivery	1700	II	28	30.5 *	22.5 *	18.2 *	3	4.1	5.1
	1700	III	54	15.9	11.7	9.5	5.8	7.9	9.8
	1700	IV	83	10.26	7.6	6.1	9	12.2	15.1
	1700	V	125	6.9	5	4	13.5	18.3	23
Maximum-pressure	1600	II	27	32 *	23 *	18.5 *	2.9	4	4.9
	1500	III	48	18	13.4	10.7	5.2	7	8.7
	1500	IV	73	11.7	8.7	7	7.9	10.7	13.3
	1500	V	110	7.8	5.8	4.7	11.9	16.1	20

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* For short-term operation only.

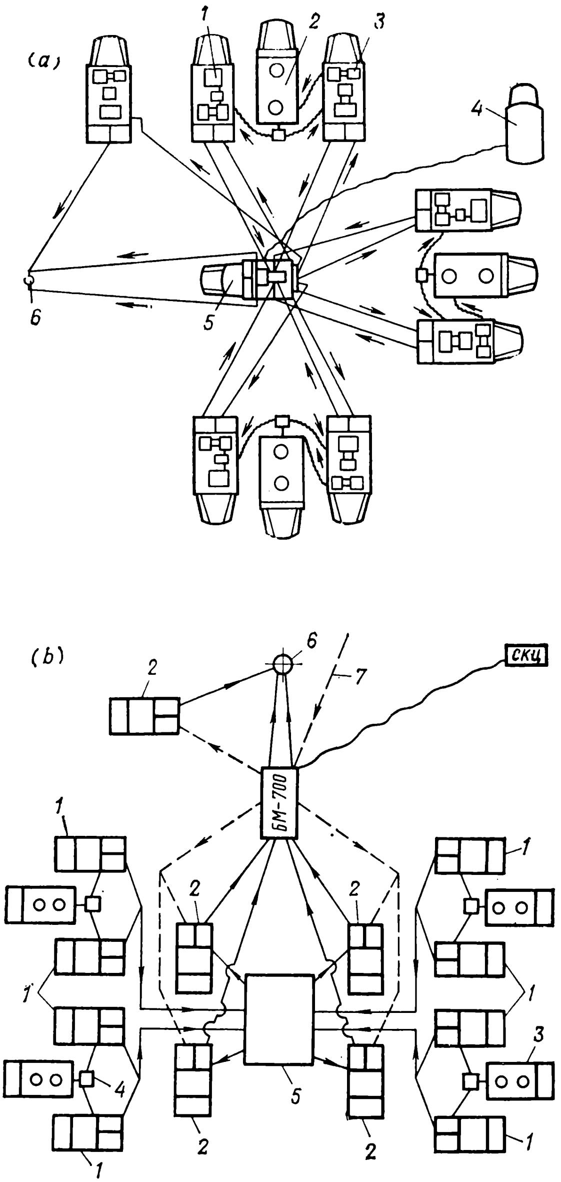

Fig. 22. Cementing equipment layouts

(*a*): *1* — 3ЦА-400А Model cementing units; *2* — 2СМН-20 Model cement mixers; *3* — ЦА-320М Model cementing units; *4* — СКЦ-2М Model cementing process control station; *5* — 1БМ-700 Model manifold unit; *6* — cementing head; (*b*): *1* — ЦА-320М Model cementing unit; 2 — 3ЦА-400А Model cementing unit; *3* — 2СМН-20 Model cement mixer; *4* — cement tank; *5* — «averaging» tank; *6* — cementing head; 7 — pipeline for delivering the displacing fluid to cementing units

The delivery (discharge) pipes of the 9T and 1B Model pumps are equipped with pressure gauges; that of the 9T Model pump is also provided with a safety valve.

A three-way cock incorporated in the intake (suction) line of the 9T Model pump makes it possible to draw fluid either from the measuring tank or from the cement tank that is, as a rule, set up on the ground. The measuring tank is equipped with bottom valves enabling either half (compartment) of the tank to be connected to a small auxiliary chamber communicating with the intake pipes of both pumps. The measuring tank compartments are provided each with a gauge rod marked every 0.1 m^3.

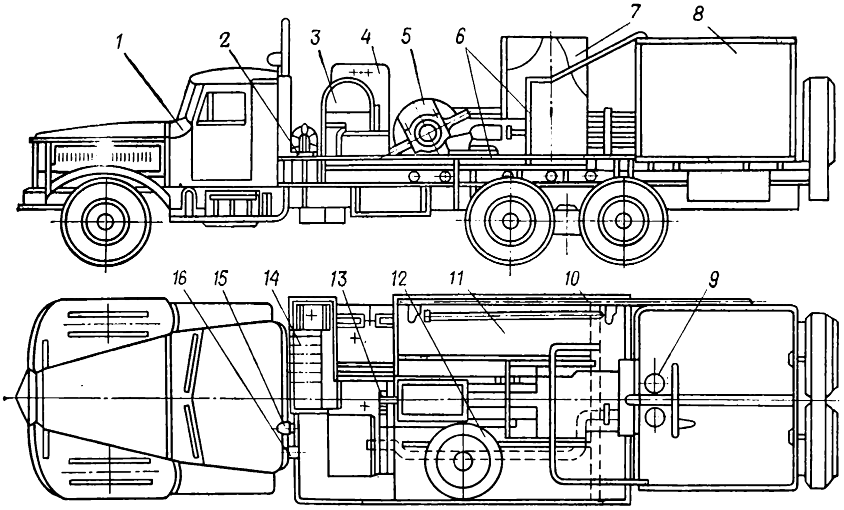

Fig. 23. ЦА-320М Model cementing unit

1 — cab; *2* — transfer case; *3* — 1B Model water pump; *4* — ГАЗ-51А Model engine; *5* — 9T Model cementing pump; *6* — piping; *7* — protective shroud of 9T Model pump; *8* — measuring tank; *9* — bottom valves; *10* — all-metal flexible hose; *11* — platform; *12* — cement mixer; *13* — universal-joint shaft; *14* — swivel elbow joints; *15* — headlamp and electrical equipment; *16* — exhaust pipe

The principal component part of the cementing unit is the 9T Model cementing pump (Fig. 24). It is a horizontal-type, two-cylinder (duplex), double-acting pump intended to force the cement slurry and drilling mud into the well being cemented.

The 1B Model water pump (Fig. 25) is a vertical, three-plunger (triplex), single-acting type and is intended for delivering a mixing fluid (e. g., water) to a vacuum-hydraulic mixing device when preparing the cement slurry.

The drive shaft of the pump receives rotation from the ГАЗ-51А Model engine through a chain coupling, and the pump crankshaft is driven by the drive shaft through a pair of spur gears.

The hydraulic part of the pump comprises a cast-iron valve chest housing three intake (suction) and three delivery (discharge) valves.

The delivery line of the cementing unit is connected to the well-head setup by means of a collapsible pipeline consisting of high-pressure pipes and swivel elbow joints.

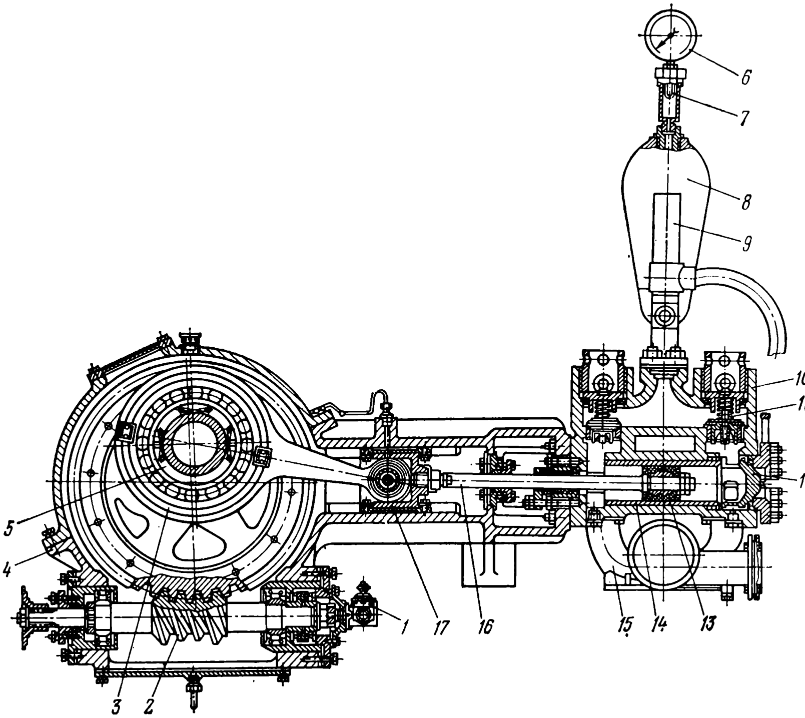

Fig. 24. 9T Model cementing pump

1 — lobe rotary pump; *2* — globoid worm; *3* — connecting rod; *4* — frame; *5* — split insert; *6* — pressure gauge; *7* — trap; *8* — delivery (discharge) manifold; *9* — safety valve; *10* — valve chest; *11* — valve; *12* — thrust cap; *13* — piston; *14* — cylinder sleeve; *15* — intake (suction) manifold; *16* — piston rod; *17* — crosshead

Water from the field water main is delivered to one of the measuring tank compartments whence it is drawn by the water pump and fed to the cement mixer. In the mixer, water is mixed with cement to form a slurry. The slurry is delivered to the cement tank from which it is drawn by the 9T Model pump and forced into the well. Once the pumping of the cement slurry into the well is completed, the water pump is stopped, the measuring tank is quickly disconnected from the water main, connected to a drilling mud line prepared beforehand, and filled with the mud by means of a mud (slush) pump.

While the mud pump is delivering the drilling mud into one of the measuring tank compartments, the 9T Model cementing pump

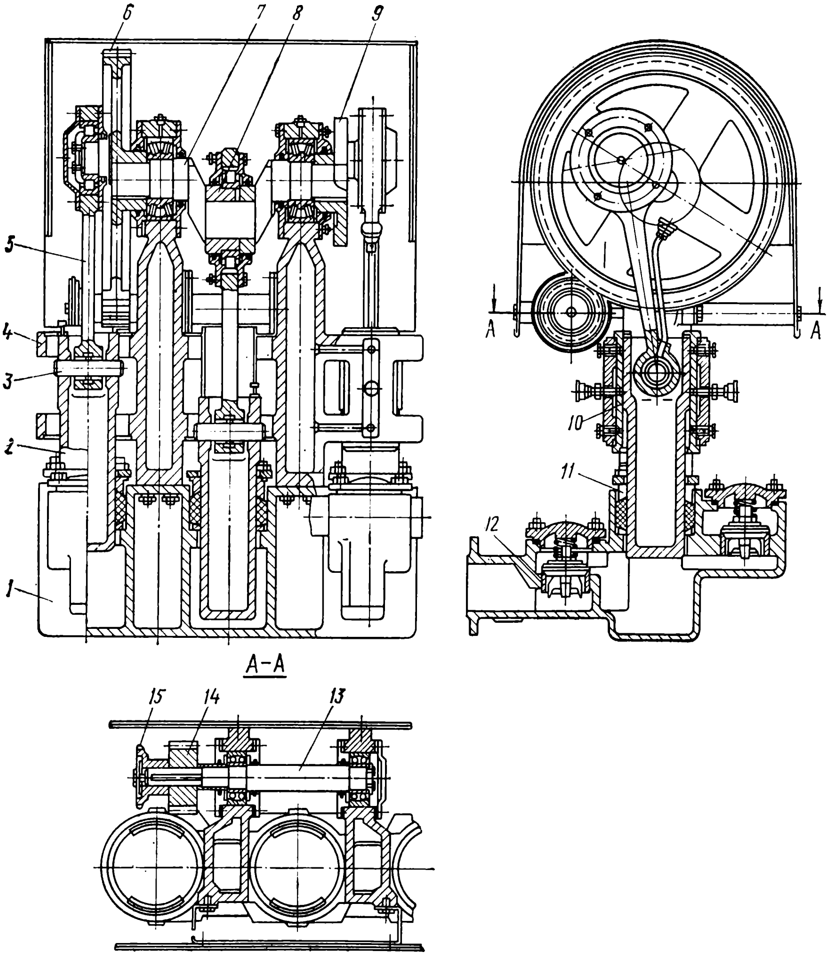

Fig. 25. 1B Model water pump

1 — valve chest; *2* — plunger; *3* — crosshead pin; *4* — frame; *5* — connecting rod; *6* — spur gear; *7* — crankshaft; *8* — split insert; *9* — crank; *10* — plate; *11* — stuffing box; *12* — valve; *13* — drive shaft; *14* — pinion; *15* — chain half-coupling

draws the mud from the other measuring tank compartment and forces it into the well. To ensure normal operation of the cementing unit, it is necessary that the measuring tank should be filled more quickly than it is emptied during a cementing job.

The 3ЦА-400А Model cementing unit (Fig. 26) is provided with a special engine and a four-speed gearbox to drive the 11T Model three-cylinder (triplex) cementing pump and is intended for pumping cement slurries and other fluids into wells during cementing, hydraulic formation fracturing, and other operations (Table 5).

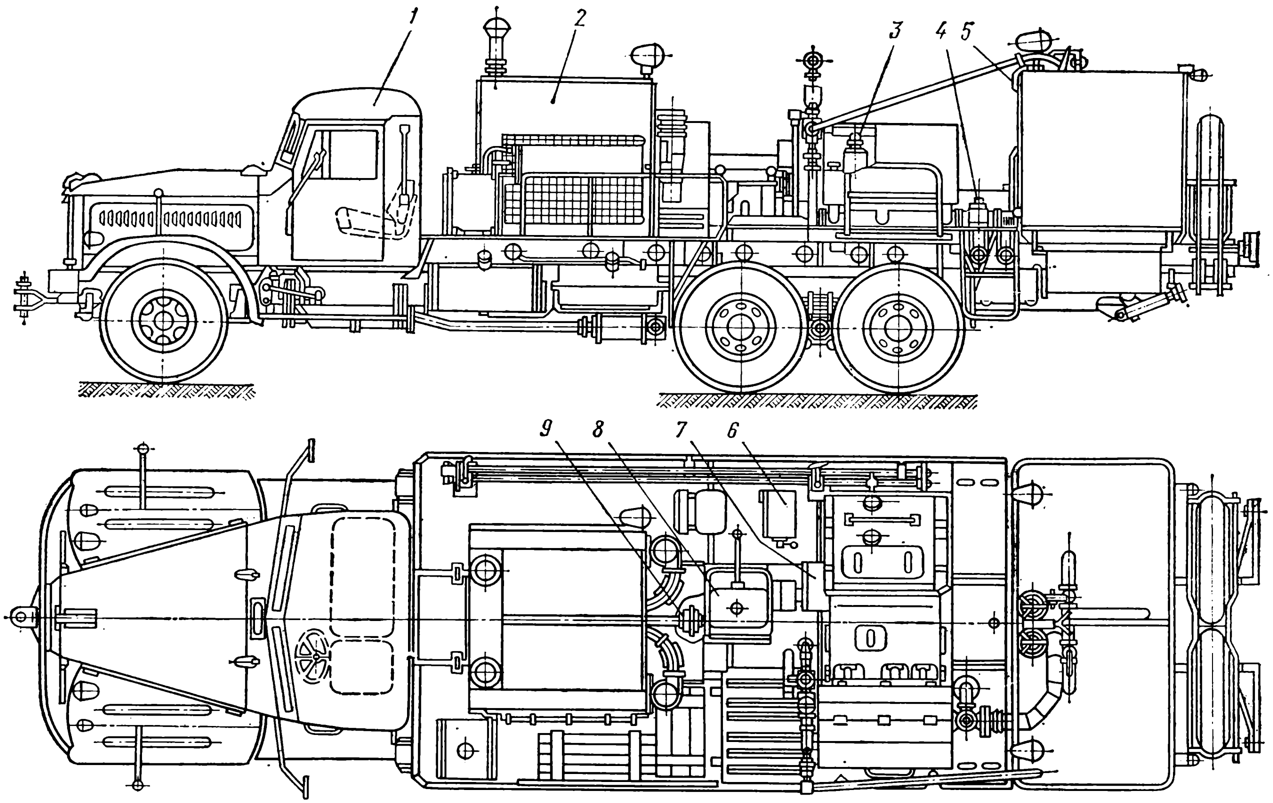

Fig. 26. ЗЦА-400А Model cementing unit

1 — cab; 2 — power plant; 3 — cementing pump; 4 — manifold; 5 — measuring tank; 6 — control station; 7 — reduction gear and clutch; 8 — gearbox; 9 — layshaft

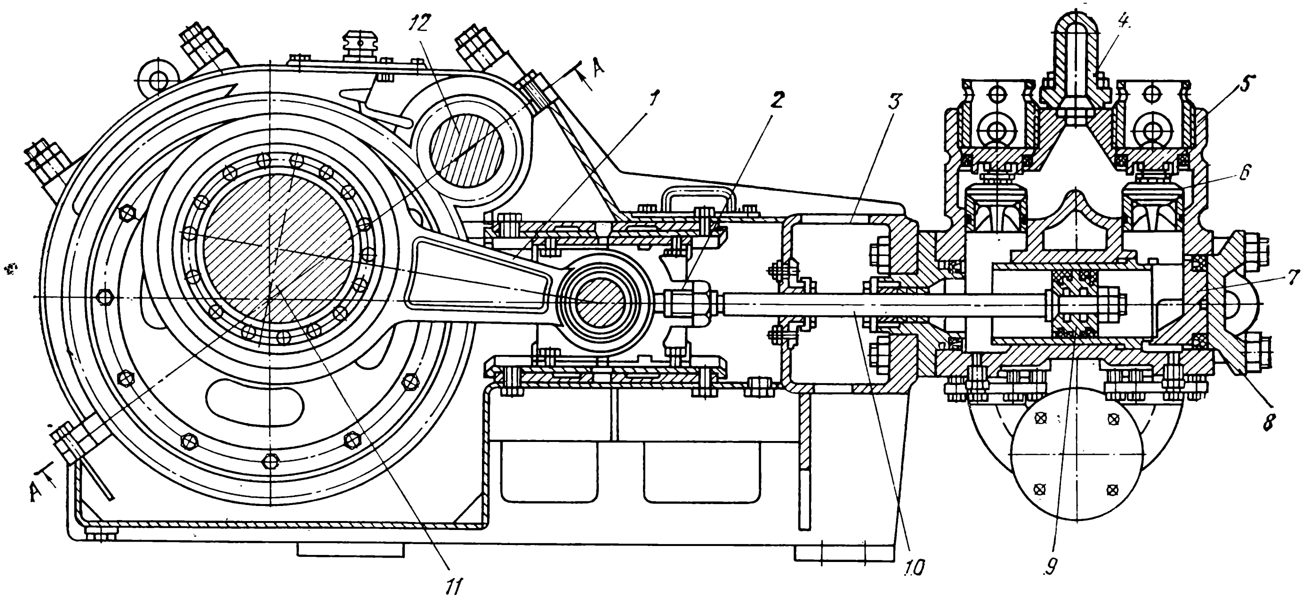

Fig. 27. 11T Model cementing pump

1 — connecting rod; *2* — crosshead; *3* — frame; *4* — manifold; *5* — valve chest; *6* — valve; *7* — thrust box; *8* — cylinder cover; *9* — piston; *10* — piston rod; *11* — crankshaft; *12* — drive shaft

Table 5

Operating Conditions of the 3ЦА-400А Model Cementing Unit

Gear engaged	Pump speed, rpm	Pressure, MPa, at cylinder sleeve diameter of, mm			Delivery, l/s, at cylinder sleeve diameter of, mm		
		110	125	140	110	125	140
I	43.2	40	30	23.5	6.6	8.8	11.2
II	62	27.5	21	16.2	9.5	12.6	16.1
III	91.8	18.5	14	11	14.1	18.6	23.8
IV	127	13.5	10	8	19.5	25.8	33

The unit consists of the following main component parts mounted on a common frame on the platform of the unit: a three-cylinder, double-acting pump, power plant, gearbox, layshaft, reduction gear, manifold assembly, and measuring tank. It has no water pump.

Main Technical Data on the 3ЦА-400А Model Cementing Unit

Type	Self-propelled, truck-mounted
Overall dimensions, mm:	
length	10 770
width	2 900
height	3 270
Total mass, fully fueled, kg	22 500
Truck:	
Model	КрАЗ-257
Load-carrying capacity, t	10 to 12
Engine:	
Model	ЯМЗ-238
Maximum speed, rpm	2 100
Maximum power, kW	177
Cementing pump:	
Model	11T
Type	Horizontal, three-cylinder, double-acting
Maximum pressure at delivery of 2.9 l/s, MPa	40
Maximum delivery at a pressure of 4 MPa, l/s	33
Hydraulic power, kW	258
Piston stroke, mm	220
Maximum number of double strokes per minute	127
Manifold assembly and other equipment:	
Measuring tank capacity, m^3	6
Nominal pipeline diameter, mm:	
intake	125
distribution	100
delivery	50
Total length of collapsible pipeline, mm	22 000
Cementing pump drive engine:	
Model	В2-500А-С2
Rated power at 1800 rpm, kW	368
Maximum torque at 1100 to 1200 rpm, N m	2250
Operating power at 1600 rpm, kW	344
Minimum idle speed, rpm	$\geqslant 600$

Gearbox:

Model	4КПМ
Maximum transmitted power at 1800 rpm, kW	368
Number of gear ratios	4
Gear ratios:	
I	4.66
II	3.26
III	2.2
IV	1

The manifold assembly of the unit includes intake and delivery pipelines equipped with plug valves 25.4, 50.8, 101.6, and 127 mm in diameter, a jiggle pin type safety valve, a discharge surge chamber, and a 60-MPa pressure gauge with an oil-filled trap.

The measuring tank 6 m^3 in capacity is divided into two equal parts and is equipped with bottom valves

The 11T Model cementing pump (Fig. 27) has three cylinders and pistons arranged horizontally.

The basis of the hydraulic part of pump are three valve chests held to the pump frame.

The pump is provided with changeable cylinder sleeves and pistons 110, 125, and 140 mm in diameter to operate at pressures of 40, 30, and 23.5 MPa, respectively.

The manifold assembly of the cementing unit includes intake, distribution, and delivery pipelines. The intake pipeline of the 11T

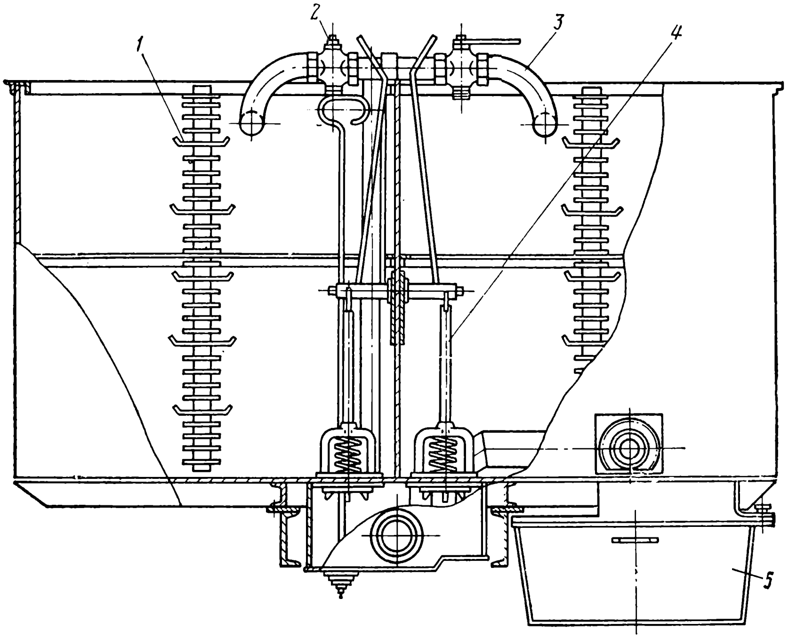

Fig. 28. Measuring tank with bottom valves

1 — measuring rod; 2 — plug valve 76.2 mm in diameter; 3 — branch pipe; 4 — bottom valves; 5 — cement tank

Model cementing pump is equipped with a three-way cock enabling fluid to be drawn either from the measuring tank of the unit or from a cement tank set up on the ground. The distribution pipeline is also fitted with a three-way cock. The delivery pipeline is provided with an air-filled discharge surge chamber, safety valve, pressure gauge with an oil-filled trap, and high-pressure plug valves.

The measuring tank (Fig. 28) is of welded construction and is installed in the rear part of the cementing unit. The purpose of the tank is to hold either a mixing fluid or a displacing fluid.

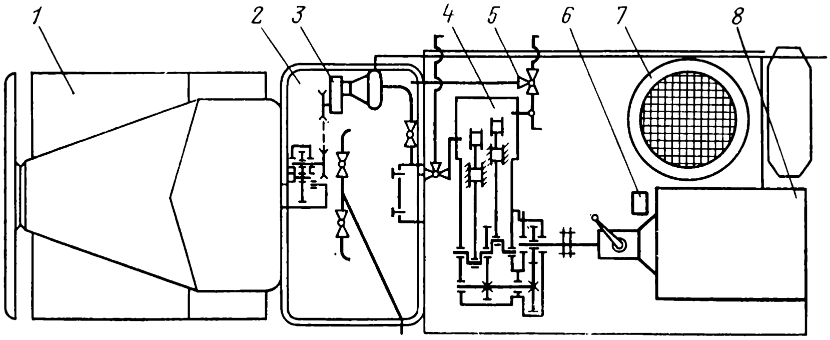

Fig. 29. 4ЦА-100 Model cementing unit

1 — truck chassis; *2* — measuring tank; *3* — water pump; *4* — cementing pump; *5* — manifold; *6* — control station; *7* — mixer; *8* — power plant

The tank is 6 m^3 in capacity and is partitioned into two equal compartments. Each compartment is equipped with a measuring rod marked every 0.1 m^3.

The measuring tank compartments are filled with fluid by a pipeline with two branch pipes passing through the tank bottom. Each branch pipe is provided with a plug valve and an elbow.

Fluid can be drawn separately from either measuring tank compartment. Sediment and wash water can be drained from the tank through a taper drain plug.

The control station of the cementing unit is arranged directly on its platform on the drive side of the cementing pump. The station includes the operator's seat, control levers and pedals, and an instrument panel.

The 4ЦА-100 Model cementing unit (Fig. 29) is mounted on the chassis of a six-wheel motor truck. The cementing pump of the unit is driven by a power plant that is installed on the unit's platform and includes a carburettor engine, five-speed gearbox, and friction clutch. The unit uses the 11ГрЦ Model two-cylinder cementing pump and the 3К-6 Model water pump driven from the truck engine.

3.2. Cementing Units of Special Construction

Specialists in this country have developed a complex of equipment for cementing wells in areas difficult of access. The complex consists of a cementing unit, cement trucks, and a mixing unit. Each piece of equipment is designed as a self-contained unit that can be used not only for cementing wells, but also for a number of other jobs. For example, the mixing unit can be used in casing cementing carried out with the aid of mud pumps, to supply water, etc.

Cementing units of this type differ from one another by the carrier and the method of transportation to the job site. They can be mounted on a frame (5ЦА-320 Model units) to be transported suspended from the МИ-6 Model helicopter, on a sledge (5ЦА-320С Model units) to be towed by a tractor, or on a tracked trailer (5ЦА-320ГБ Model units) to be towed by the С-100Б Model swamp tractor.

The 5ЦА-320 Model cementing unit consists of a power plant including an internal combustion engine and a five-speed gearbox, the 9T Model cementing pump, a measuring tank 4 m^3 in capacity, auxiliary equipment, and a control board, all mounted on a frame. The frame is provided with four pins to which slings are fastened to carry the unit suspended from the МИ-6 Model helicopter. Technical data on the 5ЦА-320 Model cementing unit are listed in Table 6.

Table 6

Technical Data on the 5ЦА-320 Model Cementing Unit

Duty	Engine speed, rpm	Gear engaged	Pump speed, rpm	Cylinder sleeve diameter, mm					
				100		115		127	
				Pump delivery, l/s	Pressure, MPa	Pump delivery, l/s	Pressure, MPa	Pump delivery, l/s	Pressure, MPa
Short-time (10% of cycle time) N = 112.2 kW	2000	II	33.6	3.56	32 *	4.75	23 *	5.95	18.5*
		III	64.2	6.8	16.8	9.08	12.6	11.36	10
		IV	97.5	10.3	11.1	13.79	8.3	17.25	6.6
		V	147.8	15.65	7.3	20.9	5.4	26.16	4.4
N = 105.6 kW	1800	II	30.2	3.2	32 *	4.27	2.3 *	5.34	18.5*
		III	57.7	6.11	17.6	8.16	13.2	10.21	10.5
		IV	87.8	9.3	11.6	12.42	8.6	15.54	6.9
		V	133	14.08	7.6	18.81	5.7	23.54	4.6
Normal N = 94.7 kW	1500	II	25.2	2.67	32 *	3.56	23 *	4.46	18.5*
		III	48.1	5.09	18.9	6.8	14.2	8.51	11.3
		IV	73.2	7.75	12.4	10.35	9.3	12.96	7.4
		V	110.9	11.75	8.2	15.69	6.1	19.63	4.9

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* For short-time operation only.

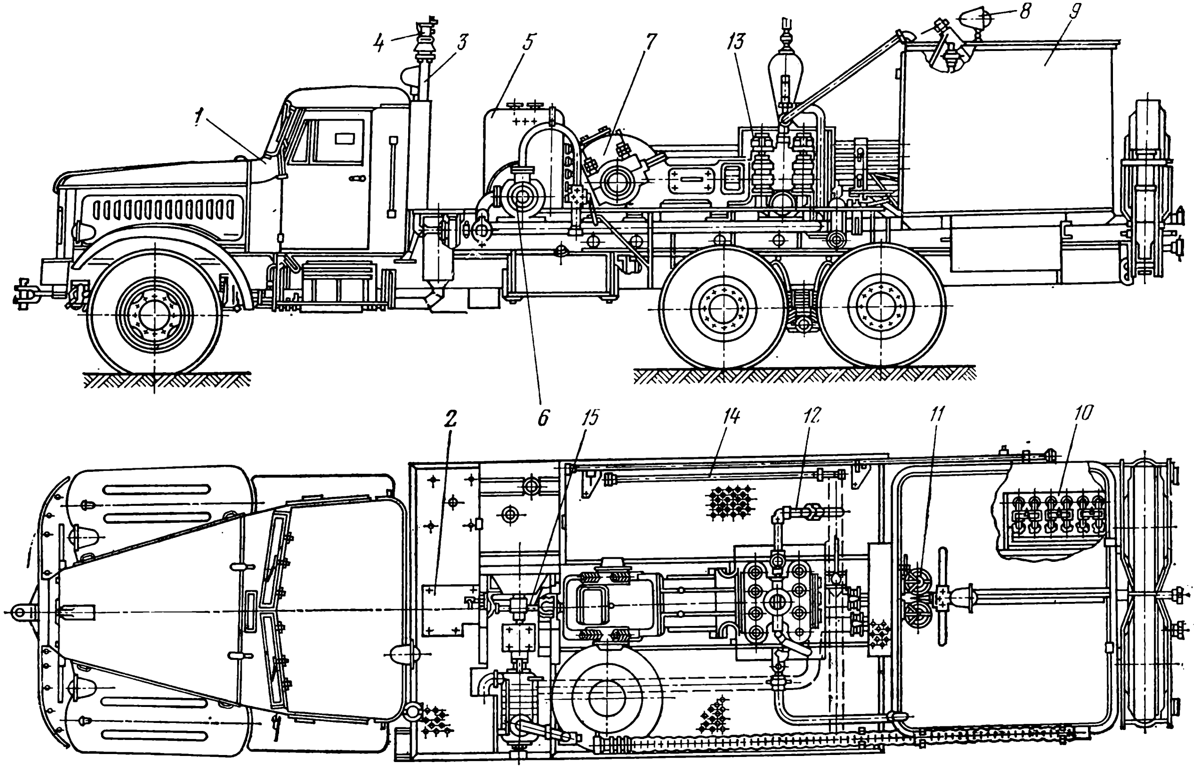

Fig. 30. ЦА-320А Model cementing unit

1 — truck chassis; *2* — transfer case; *3* — exhaust pipe; *4* — spark arrester; *5* — ГАЗ-51А Model engine; *6* — **centrifugal water pump**; *7* — 9T Model cementing pump; *8* — headlamp; *9* — measuring tank; *10* — all-metal **flexible hose** elbows; **11 — bottom valves**; *12* — **delivery** pipeline; 13 — protective pump shroud; 14 — all-metal flexible hose pipe; *15* — universal-joint shaft

The 5ЦА-320С Model cementing unit differs from the base cementer model in that it is mounted on a special sledge.

The ЦА-320А Model cementing unit (Fig. 30) differs from the ЦА-320М Model in that it uses a centrifugal water pump instead of the 1В Model plunger pump. Technical data on the ЦА-320А Model cementing unit are listed in Table 7.

Table 7

Technical Data on the ЦА-320А Model Cementing Unit

Duty	Engine speed, rpm	Gear engaged	Pump speed, rpm	Cylinder sleeve diameter, mm					
				100		115		127	
				Pump delivery, l/s	Pressure, MPa	Pump delivery, l/s	Pressure, MPa	Pump delivery, l/s	Pressure, MPa
Short-time, 10% of cycle time ($N =$ 112.2 kW)	2000	II	33.6	3.56	32 *	4.75	23 *	5.95	18.5 *
		III	64.2	6.8	16.8	9.08	12.6	11.36	10
		IV	97.5	10.3	11.1	13.79	8.3	17.25	6.6
		V	147.8	15.65	7.3	20.9	5.5	26.16	4.4
Well cementing and other short-time operations ($N =$ 105.6 kW)	1800	II	30.2	3.2	32 *	4.27	23 *	5.34	18.5 *
		III	57.7	6.11	17.6	8.16	13.2	10.21	10.5
		IV	87.8	9.3	11.6	12.42	8.6	15.54	6.9
		V	133	14.08	7.6	18.81	5.7	23.54	4.5
Long-time ($N =$ 94.7 kW)	1500	II	25.2	2.67	32 *	3.56	23 *	4.46	18.5 *
		III	48.1	5.09	18.9	6.8	14.2	8.51	11.3
		IV	73.2	7.75	12.4	10.35	9.3	12.96	7.4
		V	110.9	11.75	8.2	15.69	6.1	19.63	4.9

Notes. 1. Volumetric efficiency $\mu=0.9$. 2. To drive 9T Model cementing pump with gearbox in low gear is strictly forbidden 3. Asterisked are pressures permissible for short-time operation only.

3.3. Improvement of Cementing Units

The main disadvantages of the cementing units now in use are as follows: insufficient hydraulic power, inadequate durability of individual assemblies (valves, pistons and cylinder sleeves, seals, etc.), heavy pressure fluctuations, inadequate reliability of shut-off devices and all-metal flexible hoses, incapability of operating at low ambient temperatures, inadequate cross-country capability of truck chassis, and some others.

At present, in the USSR there has been developed a size series of cementing units and cement mixers. The pertinent USSR standard provides for the manufacture of three types of pumping units: with a measuring tank (УНБ), with a tank for transporting the working fluid (УНЦ), and without special equipment (УН). The pumping units with a measuring tank (cementing units) will be manufactured in five hydraulic powers (73.6, 117.8, 294.4, 463.7, and 910 kW).

For pumping units of one and the same useful power to have various characteristics, provision is made for using different hydraulic assemblies in the units. Several modifications of pumping units for 117.8, 294.4, and 463.7 kW have already been developed and tested. Main technical data on the УНБ Type pumping units are listed in Table 8.

Table 8

Main Technical Data on the УНБ Type Pumping Units

Number of pumps	Useful power, kW	Maximum-pressure operation		Maximum-delivery operation	
		Maximum discharge pressure, MPa	Ideal delivery, l/s	Ideal delivery, l/s	Discharge pressure, MPa
1	74	16	4.7	12.5	6
		20	3.7	10	7.5
		25	3	8	9.4
	118	25	4.7	25	4.8
		32	3.7	20	6
		40	3	16	7.5
		50	2.4	12.5	9.6
		63	1.9	10	12
			7.5		7.5
			5.9		9.4
	294		4.7	40	12
			3.7	32	15
			3	25	18.7
2	463	40	11.8	20	
		50	9.4	16	
		63	7.5		11.8
		80	5.9		14.8
		100	4.7		19
			23	80	29.6
			19	64	
	917		15	50	
			11.8	40	
			9.4	32	

Technical Data on the УНБ2-630 Model Pumping Unit

Useful power, kW	434.2
Maximum pressure, MPa	70
Delivery at maximum pressure, l/s . . .	4.8
Maximum delivery, l/s	36
Pressure at maximum delivery, MPa	12

Cementing pump type	Horizontal, three-plunger, single-acting, with two changeable hydraulic assemblies for pressures of 32 and 70 MPa
Changeable plunger diameters, mm	80 (for $p = 100$ MPa) 90, 110, 115, and 140
Plunger stroke, mm	160
Maximum number of double strokes per minute	283
Water pump:	
Model	ЦНС60-198
Type	centrifugal
Delivery, l/s	16.6
Maximum pressure, MPa	1.98
Booster pump:	
Model	5ГруЛ-12
Type	centrifugal
Delivery, l/s	41.6
Maximum pressure, MPa	0.165
Overall dimensions, mm:	
length	10 420
width	2 900
height	3 500
Mass, kg	22 160

3.4. Cement Mixers

Cement mixers are special machines intended for preparing various cement slurries and plugging mixtures used in cementing wells; they can also be used for preparing standard-density and weighted drilling muds from mud (gel) powders.

These machines help mechanize labour-consuming operations, improve the quality and stability of the slurries being prepared, reduce losses of cement, and better labour conditions of attending personnel.

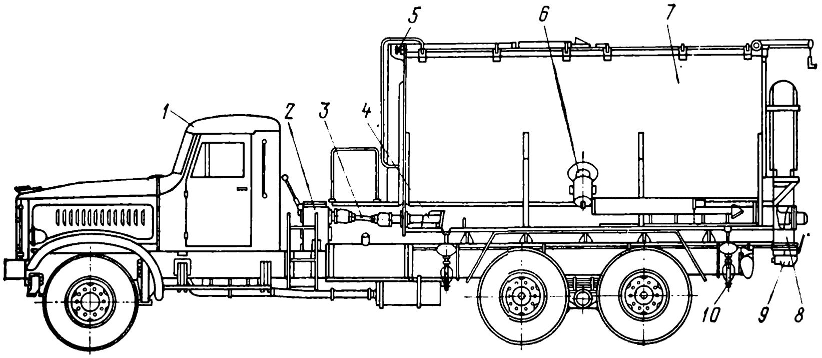

Fig. 31. 2CMH-20 Model cement mixer

1 — truck driver's cab; *2* — transfer case; *3* — universal-joint shaft; *4* — discharge (metering) screw conveyers; *5* — loading screw conveyer drive; *6* — loading screw conveyer; *7* — hopper; *8* — mixer cone; *9* — vacuum-hydraulic mixing device; *10* — folding jack

In accordance with their purpose and character of work, cement mixers are mounted either on motor trucks or trailers.

The principal assemblies of cement mixers are a hopper, loading and discharge mechanisms, and a mixing device for preparing cement slurries and other mixtures.

The 2CMH-20 Model cement mixer (Fig. 31) is designed to carry 9 t of dry cement. On arrival at the destination, the machine is supported by folding jacks and loaded to full capacity by means of a detachable vertical loading screw conveyer.

Technical Data on the 2CMH-20 Model Cement Mixer

Load-carrying capacity (running), t	8 to 9
Hopper capacity, m^3	14.5
Delivery when preparing cement slurry, l/s	20
Density of fluid being prepared, g/cm^3:	
cement slurry	1.7 to 2.1
cement-sand slurry	1.9 to 2.3
cement-bentonite slurry	1.4 to 1.6
clay drilling mud	1.02 to 1.4
weighted clay drilling mud	1.35 to 2.3
Fluid pressure in mixing device inlet line, MPa	0.8 to 1.5
Mixing device type	Vacuum-hydraulic
Maximum power consumption, kW	29.4
Loading screw conveyer delivery, t/h	12 to 15
Mass, kg	13 500
Overall dimensions, mm:	
length	9580
height, loaded	3510
height, unloaded	3550
width in transport position	2700
width in working position	4000

The amount of cement delivered to the mixing device is controlled by adjusting the speed of two horizontal metering screw conveyers. The loading of the cement mixer to capacity at the job site is effected by means of a special loading screw conveyer operating at a rate of 12 to 15 t/h by a pneumatic-discharge cement truck at a rate of 1 t/min.

The mixer cone serves to receive the cement delivered by the two screw conveyers and direct it to the mixing device. A partial vacuum created in the mixing device by a jet of fluid flowing through it causes the cement to be sucked in the device where it is vigorously mixed with the fluid in the turbulent stream in the discharge pipe.

The mixing device (Fig. 32) consists of a receiving pot that gradually transits into the discharge pipe and a choke chamber with a changeable orifice. The pot is fabricated from a light-weight alloy and is flange-fitted to the mixer cone.

Operating conditions of the 2CMH-20 Model cement mixer are given in Table 9.

Table 9

Operating Conditions of the 2CMH-20 Model Cement Mixer when Preparing Plugging Mixtures from Various Dry Materials

Plugging material	Density, g/cm^3		Water-cement ratio	Orifice diameter	Mixing water pressure, MPa	Gear engaged	Theoretical delivery, l/s
	dry material	mixture prepared					
Lightened portland cement for "cold" wells	2.65-2.73	1.42-1.50	0.9-1.05	14-16	1.0-1.2	II	17-20
Ditto, for "hot" wells	2.73-2.81	1.50-1.60	0.9-1.05	14-16	1.0-1.2	II	16-20
Portland cement for both "cold" and "hot" wells	3.12-3.15	1.82-1.85	0.45-0.5	12-14	1.0-1.5	II-III	10-13
Sand portland cement for "cold" wells	2.99-3.02	1.82-1.85	0.45-0.5	12	1.0-1.5	II-III	9.5-11.5
Ditto, for "hot" wells	2.89-2.90	1.82-1.85	0.45-0.5	12	1.0-1.5	II-III	9.5-11.5
УЦГ-1 Grade weighted cement	3.45-3.55	2.05-2.15	0.45-0.5	10	2.5-3.0	III	8.5-10.5
УЦГ-2 Grade weighted cement	3.55-3.65	2.16-2.25	0.32	10	2.5-3.0	III	8.5-10.0
Weighted slag cement:							
Grade УШЦ1-120	3.45-3.55	2.05-2.15	0.35	10	2.5-3.0	III	8.5-10.5
Grade УШЦ2-120	3.55-3.65	2.16-2.25	0.32	10	2.5-3.0	III	8.0-10.0
Grade УШЦ1-200	3.45-3.55	2.05-2.15	0.35	10	2.5-3.0	III	8.5-10.5
Grade УШЦ2-200	3.55-3.60	2.16-2.25	0.32	10	2.5-3.0	III	8.0-10.0
Jointly ground slag-sand cement Grade ШПЦС-120	280	1.78-1.82	0.45	12-14	1.0-1.5	II-III	10-13.5
Ditto, Grade ШПЦС-200	280	1.78-1.83	0.42	12-14	1.0-1.5	II-III	10-13.5

The 1CM-10 Model cement mixer (Fig. 33) has a smaller hopper capacity than the 2CMH-20 Model. It is also simpler in design: there is no loading device or folding jacks. The load-carrying capacity of this machine is 10 t. It is loaded at mechanized cement stores through hatches at the top of the hopper.

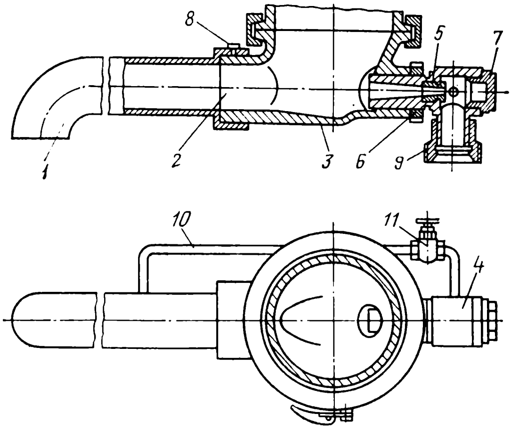

Fig. 32. Vacuum-hydraulic mixing device
1 — discharge pipe; *2* — diffuser; *3* — receiving pot; *4* — choke chamber; *5* — changeable orifice; *6* — nut; *7* — plug; *8* — set bolt; *9* — union nut; *10* — bypass pipe; *11* — valve

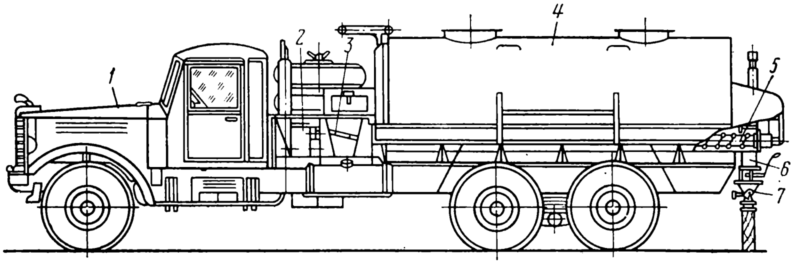

Fig. 33. 1CM-10 Model cement mixer
1 — truck chassis; *2* — transfer case; *3* — universal-joint shaft; *4* — hopper; *5* — metering screw conveyer; *6* — mixer cone; *7* — mixing device

The 1AC-20 Model cement mixer (Fig. 34*a*) is intended for holding and carrying dry plugging materials and preparing plugging mixtures independently of cementing units. It consists of a water-supply unit, a vacuum-hydraulic mixing device, and a manifold to deliver mixing water to the device. The water-supply unit is similar to that of the ЦА-320M Model cementing unit and includes the 1B Model three-plunger water pump driven by the ГА3-51A Model engine through a gearbox.

Main Technical Data on the 1AC-20 Model Cement Mixer

Load carrying capacity (running), t	8
Cement holding capacity, t	20
Delivery, l/s	20
Prepared slurry density, g/cm^3	1.7-2.0 *
Overall dimensions, mm:	
length	9580
height, loaded	3510
height, unloaded	3550
width in transport position	2800
width in working position	4000
Mass, kg:	
with fuel and two operators	15 235
without fuel	15 070
Mass distribution without load, kg:	
on front axle	4825
on rear bogie	10 410
Mass distribution with a load of 80 kN, kg:	
on front axle	4825
on rear bogie	18 410
Control	Centralized, from truck driver's cab
Water pump:	
Model	1B
Type	Vertical, three-plunger, single-acting
Plunger diameter, mm	125
Plunger stroke, mm	170
Drive gear ratio	3.88
Maximum speed, rpm	140
Delivery l/s	13
Pressure, MPa	1.5
Power consumption, kW	25.8
Manifold:	
Intake line diameter, mm	100
Delivery line diameter, mm	50

The 2AC-20 Model cement mixer (Fig. 34*b*) serves to carry and hold dry plugging materials and to prepare cement slurries, clay drilling muds, and sand-fluid mixtures independently of cementing units.

This mixer differs from the 1AC-20 Model in that it employs the 4K-6 Model centrifugal water pump instead of the 1B Model three-plunger pump. The 4K-6 Model pump is driven by the truck engine via a transfer case and a universal-joint shaft.

The CMП-20 Model cement mixer (Fig. 35) is mounted on a trailer 20 t in load-carrying capacity. The hopper of this machine has a more rigid frame than the 2CMH-20 Model mixer. The discharge screw conveyers here are longer than those of the 2CMH-20 and CM-10 Models and rotate in different directions, the left-hand screw conveyer being provided with a left-hand thread and the right-hand conveyer, with a right-hand thread, which makes for better conveyance of cement along the hopper

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* Weighted plugging mixtures may have higher density values.

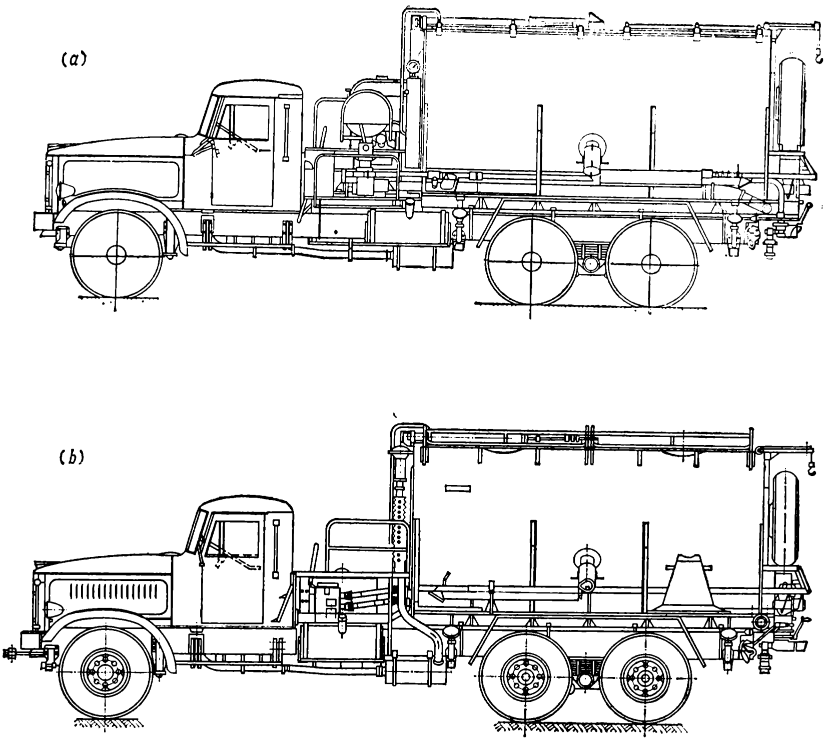

Fig. 34. Cement mixers
(*a*) Model 1AC-20; (*b*) Model 2AC-20

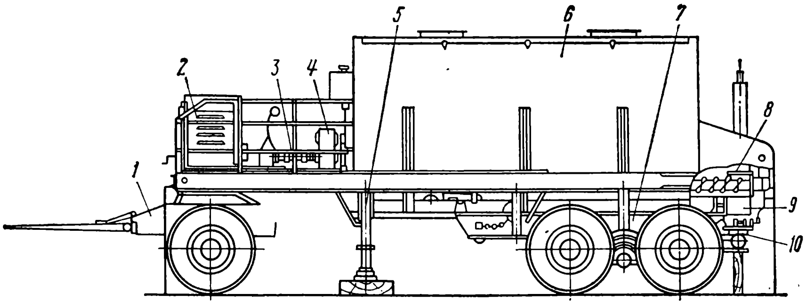

Fig. 35. СМП-20 Model cement mixer
1 — trailer; *2* — engine; *3* — shaft; *4* — gearbox; *5* — jack; *6* — hopper; *7* — frame; *8* — discharge screw conveyer; *9* — mixer cone; *10* — mixing device

bottom. Technical data on the СМП-20 Model cement mixer are similar to those of the 2СМН-20 Model.

The CM-4M Model cement mixer is intended for carrying dry cement and preparing cement slurries. It consists of a hopper, metering (discharge) screw conveyer with a drive assembly, and instrumentation (Fig. 36).

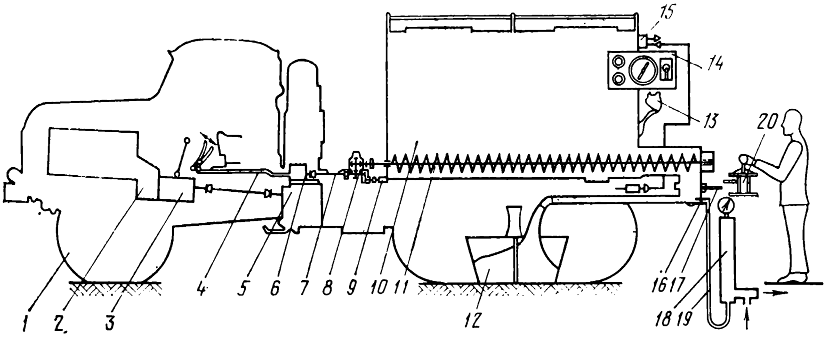

Fig. 36. Illustrating the operation of the CM-4M Model cement mixer

1 — ЗИЛ-131А Model truck chassis; *2* — truck engine; *3* — gearbox; *4* — power takeoff shifter rod; *5* — transfer case; *6* — power takeoff case; *7* — universal-joint shaft; *8* — reduction gear; *9* — tachometer transducer; *10* — hopper; *11* — screw conveyer; *12* — cement tank of cementing unit; *13* — swiveling headlamp with switch; *14* — instrument panel with tachometer, ignition switch, and two pilot lamps; *15* — C-80 Model pneumatic vibrator; *16* — changeable orifice; *17* — mixer cone gate; *18* — pressure regulator; *19* — bypass pipe; *20* — back-up control lever

Recommended operating conditions for the CM-4M Model cement mixer are presented in Table 10.

Table 10

Recommended Operating Conditions for the CM-4M Model Cement Mixer to Prepare a Cement Slurry 1.85 g/cm³ in Density

Delivery, l/s	Speed, rpm	Pressure, MPa, upstream of mixing device at orifice diameter of, mm					Water consumption, l/s
		11	12.35	13.5	14.6	15.6	
6.75	61	1	0.65	0.45	—	—	4.1
8.25	76		0.7	0.7	0.52	—	5.15
10	91		1	1	0.74	0.54	6.25

The hopper of the CM-4M Model cement mixer is a metal vessel with inclined side walls and a trough-shaped bottom accommodating a discharge screw conveyer. The inner surfaces of the hopper front and rear walls bear three marks each. The lower mark corresponds to a hopper capacity of 1 m³, the middle mark — 2 m³, and the upper mark — 3 m³. The hopper is held to the truck frame by means of clamps and brackets.

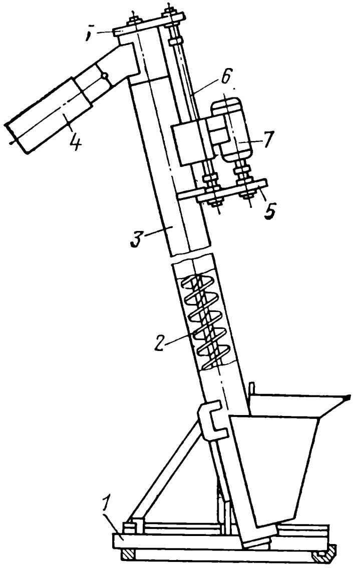

Fig. 37. ПШ Model loading screw conveyer

1 — base; *2* — screw; *3* — pipe; *4* — swiveling pipe; *5* — reduction gear; *6* — universal-joint shaft; *7* — electric motor

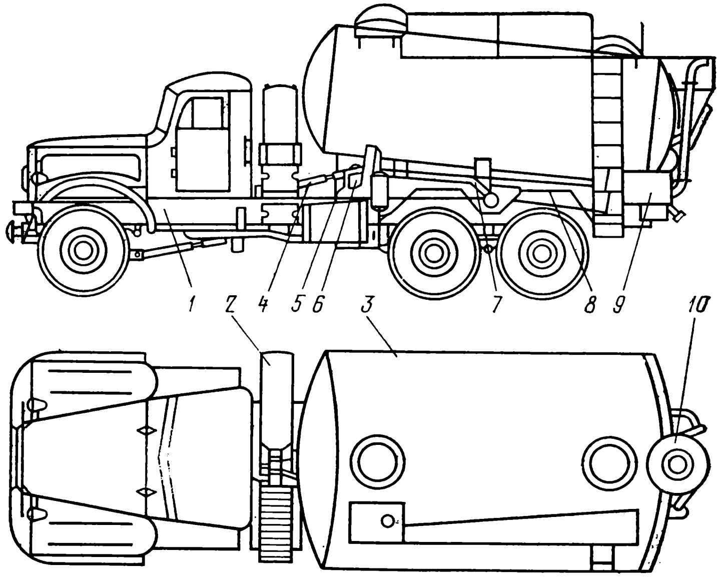

Fig. 38. ЗАС-30 Model cement mixer

1 — truck driver's cab; *2* — transfer case; *3* — tank; *4* — transmission; *5* — rotary compressor; *6* — gear pump; *7* — manifold; *8* — pneumatic discharge system; *9* — control station; 10 — trap

The ПШ Model loading screw conveyer (Fig. 37) is used for loading cement mixers directly at the job site. The conveyer is equipped with a loading hopper at the bottom and a swivelling discharge pipe with a fabric sleeve at the top.

The 3AC-30 Model cement mixer (Fig. 38) is intended for carrying and holding dry cement and preparing cement slurries. It differs from the other Soviet-made cement mixers mainly in that it uses a pneumatic dry-cement handling system instead of a mechanical one.

The mixer consists of a tank mounted on the chassis of the КрАЗ-255Б Model cross-country motor truck, a compressor and water pump driven from the truck engine through a transfer case, a transmission, manifold, discharge system, vacuum-hydraulic mixing device, and a control station.

Technical Data on the 3AC-30 Model Cement Mixer

Load-carrying capacity (running), t	6 to 7
Cement holding capacity, t	20
Delivery, l/s	30
Prepared slurry density, g/cm^3	1.3 to 2.4
Discharge system type	Pneumatic
Discharge rate, t/min	2.2
Rotary compressor:	
Model	РК-6/1
Delivery, m^3/min	6
Maximum pressure, MPa	0.25
Tank pressure, MPa	0.06
Water pump:	
Model	4К-6
Delivery at a pressure of 2 MPa, l/s	2.5
Power consumption, kW	45.2 to 55.2
Overall dimensions, mm:	
length	9200
width	2850
height	3680
Mass (running), fully loaded, kg	19 675

The tank discharge system is located at the rear of the tank, therefore the tank is tilted backwards at 6° to the horizontal. At the bottom of the tank there are two airslides that serve to fluidize the bottom layer of dry cement and make it flow during its discharge from the tank with the aid of compressed air. Each of the two discharge pipes of the tank is equipped with a gate to shut off the exit for cement when purging the discharge line. The discharge pipes are also provided with doors to remove cement cake and stones.

For the prepared slurry to have a more uniform density, provision is made to maintain a certain level of cement in the mixer cone.

The manifold serves to receive the mixing fluid from the measuring tanks of cementing units or from other devices and deliver it to the vacuum-hydraulic mixing device.

Concentrated at the control station at the rear of the cement mixer are instrumentation and levers to control the mixer mechanisms.

The loading system of the cement mixer provides for mechanized loading of cement delivered to the job site in sacks. The system uses the compressor, a loading hopper, and a loading pipe located on the left-hand side of the tank. The self-loading of cement is effected using the vacuum created in the tank by the rotary compressor.

3.5. Cementing Process Control Station and Self-Propelled Manifold Unit

The СКЦ-2М Model cementing process control station (Fig. 39) is intended for controlling the main parameters of cement slurry and conditions of its pumping into the well being cemented. The station includes a self-propelled laboratory accommodated in the body of the КАВЗ Model truck and housing secondary and auxiliary instruments and the 1БМ-700 Model self-propelled manifold unit mounted on the chassis of the ЗИЛ-131 Model truck and consisting of delivery and distribution manifolds, a collapsible pipeline, and a set of transducers.

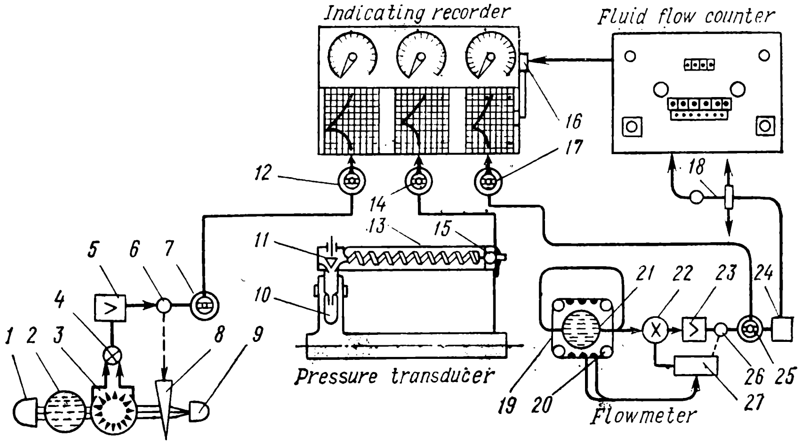

Fig. 39. СКЦ2-М Model cementing process control station

1 — radioactive source; *2* — density transducer pipe section; *3* — receiving counter rotating drum; *4* and *22* — comparison elements; *5* and *23* — amplifiers; *6* and *26* — servomotors; *7*, *15*, and *25* — transmitting synchros; *8* — circular compensation wedge; 9 — small compensating radioactive source; *10* — trap; *11* — adjusting choke; *12* — densimeter receiving synchro; *13* — helical tube; *14* — pressure gauge receiving synchro; *16* and *17* — flowmeter receiving synchros; *18* — two-position clutch-motor; *19* — flow rate transducer; *20* — self-balancing potentiometer feedback supply system; *21* — electrode; *24* — converter; *27* — feedback insertion element

The instruments of the station help control and record the following main cementing process parameters: the pressure, flow rate, total volume, and density of the fluid pumped into the well, The СКЦ-2М Model station can be employed when carrying out hydraulic fracturing of formations and also other processes involving the use of electrically conductive media at pressures up to 40 MPa.

The density of the slurry being pumped into the well is measured by means of the ПЖР-2М Model radioactive densimeter. The operating principle of the densimeter is based on the absorption of a gamma-ray beam on its passage through a fluid layer. The intensity of absorption of these rays changes in accordance with the density of the fluid.

Technical Data on the СКЦ-2М Model Cementing Process Control Station

Measurement range:	
pressure, MPa	0 to 40
flow rate, l/s	5 to 100
density, g/cm^3	1 to 2
Fluid meter capacity, m^3	9999
Basic measurement error referred to full-scale value, %:	
pressure	±2.5
flow rate	±2.5
throughput fluid volume (relative error)	±2.5
density (for 1-g/cm^3 measurement range)	±3.0
Indicating recorder chart speed, mm/h	600
Power supply (a.c. mains or self-contained source):	
voltage, V	220±10%
frequency, Hz	50±4%
Power consumption, W	800

The 1БМ-700 Model self-propelled manifold unit (Fig. 40) serves to connect the delivery pipelines of cementing units to the wellhead equipment and also to distribute the displacing fluid among the units during cementing.

Technical Data on the 1БМ-700 Model Self-Propelled Manifold Unit

Delivery manifold:	
maximum operating pressure, MPa	70
nominal inside diameter, mm	100
number of connected pipelines from cementing units	6
number of connected pipelines to wellhead equipment	2
nominal inside diameter of pipelines, mm	50
Distribution manifold:	
maximum operating pressure, MPa	2.5
nominal inside diameter, mm	100
number of connected pipelines	10
nominal inside diameter of connected pipelines, mm	50
Collapsible pipeline:	
number of pipes	23
total pipeline length, m	80
nominal inside diameter, mm	50
number of swivelling elbow joints	16
Overall dimensions, mm:	
length	7690
width	2500
height	2895
Mass, with fuel and two operators, kg	8440

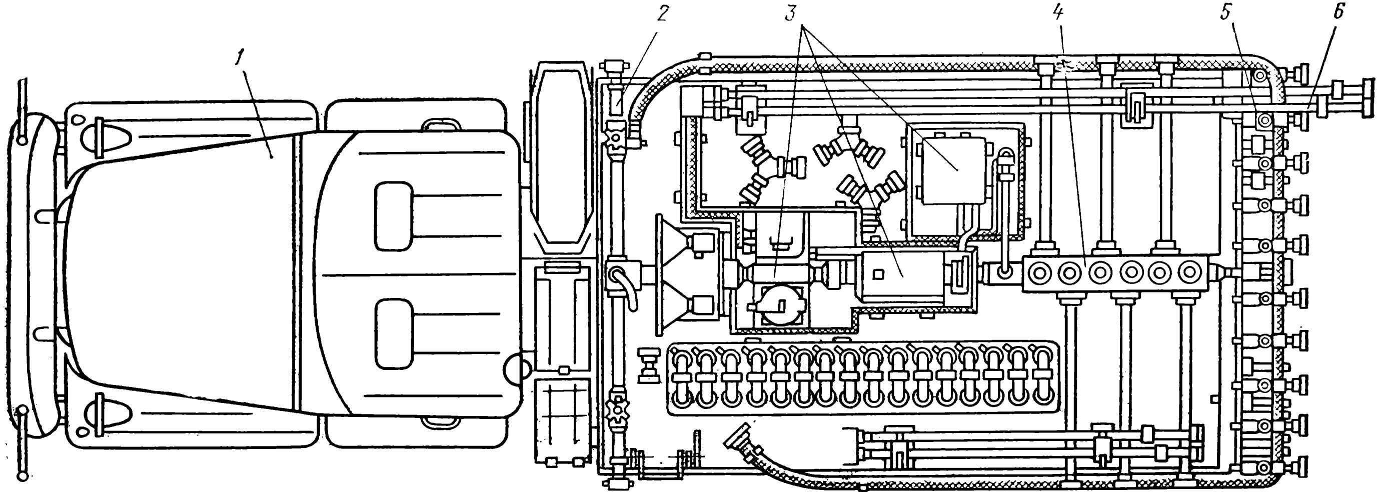

Fig. 40. 1БМ-700 Model self-propelled manifold unit

1 — truck chassis; *2* — delivery line; *3* — density, flow rate, and pressure transducers; *4* — delivery manifold; *5* — distribution manifold; *6* — collapsible pipeline

The delivery manifold includes a valve chest with six branch pipes for connecting the delivery pipelines of cementing units and a pipe 100 mm in nominal inside diameter carrying the transducers of the cementing process control station. The pipe terminates in a tee piece with one branch connected to a safety valve and the other two, to pipelines leading to the wellhead equipment.

The distribution manifold is a pipe 100 mm in nominal inside diameter to which are welded 10 nipples, each equipped with a plug valve having a screwed-in taper connector for attaching collapsible pipelines.

Connecting the so-called "forks", which are included as a standard equipment of the manifold unit, either to the delivery or to the distribution manifold enables one to increase the number of connected pipelines from 6 to 10 or from 10 to 14, respectively.

The self-propelled manifold unit is equipped with a swinging boom 400 kg in lifting capacity for handling various devices and pipes and fittings that are usually carried on the platform of the unit.

When operating the manifold unit, one must strictly adhere to all safety regulations, for the high fluid pressures involved in the cementing process are dangerous. Moreover, there is a radioactive source on the unit.

3.6. Cementing Process Calculations

Calculations pertaining to the process of cementing wells are an important part of planning this final stage of well construction. However, an exact calculation of the entire cementing process is as yet impossible, because there are no true data on the rheological properties of slurries and the size and configuration of the well bore, and local hydrodynamic resistances in the well are difficult to determine. Therefore, resort is made to a stochastic (rather than deterministic) approach to the hydraulic process of well cementing.

In accordance with concrete local conditions, particular calculation methods are being developed, based mainly on the magnitude of hydraulic fracturing pressure of formations.

Calculation of Single-Stage Casing Cementing Process

The calculation of a single-stage casing cementing process includes:

(1) determination of the amount of dry cement (or of a mixture of cement with aggregates) and selection of the type of cement;

(2) determination of the required amount of water and reagents for mixing the plugging cement and determination of the amount of the displacing fluid;

(3) determination of the permissible cementing time, and selection of the plugging cement formulation;

(4) determination of the required number of cementing units and cement mixers.

The maximum possible pressure at the end of cementing is sometimes determined.

1. Given the static bottom-hole temperature $t_{s.\,bh}$, the dynamic (flowing) bottom hole temperature $t_{d.\,bh}$ (during flushing) is determined by the following formula:

$$t_{d.\,bh.} = t_{s.\,bh.} + t_{wh.\,0} - t_{wh} - \Delta t_f \tag{7}$$

where $t_{wh.\,0}$, t_{wh} is the temperature of the "neutral" ground layer and of the drilling mud at the wellhead, respectively, during flushing fluid circulation after 1-2 cycles; Δt_f is the difference in temperature between the drilling mud flowing out of the well and the drilling mud pumped into the well.

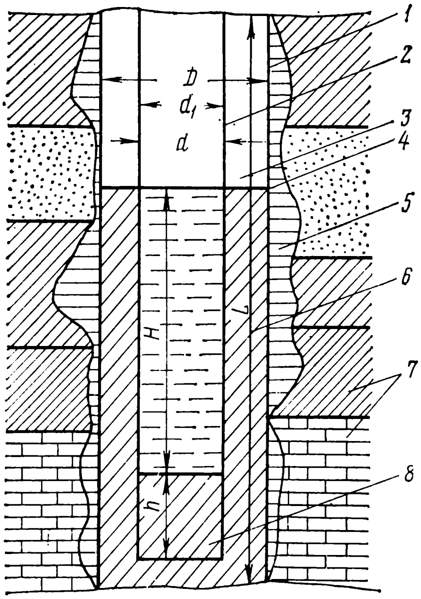

Fig. 41. Illustrating calculation of cementing process

1 — nominal well bore diameter; 2 — casing string; 3 — drilling mud; 4 — plugging mixture; 7 — rocks; 8 — cement plug

At the bottom-hole temperatures of up to approximately 50 °C use is made of cements for "cold" wells; cements for "hot" wells are employed when the bottom hole temperature equals about 50-90 °C, and those for deep wells (sand cement, blast-furnace slag cement) are applied at higher temperatures.

Having selected the type of the plugging cement and knowing its physical characteristics, the required amount of it is then determined.

2. Figure 41 shows a diagram of a well. The volume $V_{c.\,s}$ of cement slurry required to cement a well interval of height H is equal to the volume of the annular space of the same height H, with due regard for the cavernosity ratio k, plus the volume of the cement plug left in the well:

$$V_{c.\,s.} = \frac{\pi}{4}\left[k\,(D^2 - d^2)\,H + d_1^2 h\right] \tag{8}$$

or

$$V_{c.\,s.} = (V - V_c) + 0.785\,dh \tag{8'}$$

where D, d, and d_1 are the well bore diameter, outside casing diameter, and inside casing diameter, respectively, m; h is the height of the cement plug left in the well (i. e., the distance from the casing shoe to the cement baffle collar), m; V is the well bore volume within the cementing interval, determined on the basis of section gauge logging data, m^3; V_c is the casing string outside volume within the cementing interval, m^3.

The cavernosity ratio k usually varies from 1.2 to 2.5. When making calculations for cementing, it is preferable to use the cavernosity ratio values established by the geological service for particular wells, or to take as a guide the data available for already cemented wells.

Table 11

Density of Plugging Cements

Cement Grade	ρ_c, g/cm^3	Cement Grade	ρ_c, g/cm^3
For "hot" wells	3.1	УШЦ1-200	3.4
УЦГ-1	3.5	УШЦ2-200	3.6
УЦГ-2	3.7	ШПЦС-120	2.8
УШЦ1-120	3.4	ШПЦС-200	2.8
УШЦ2-120	3.6	Slag	2.9

3. To prepare 1 m^3 of cement slurry of a given water-cement ratio m (usually equal to 0.5) by using water of density ρ_w, it is necessary to take q tons of cement of ρ_c (Table 11):

$$q = \frac{\rho_c \rho_w}{\rho_w + m\rho_c} \tag{9}$$

Then the density of the slurry will be

$$\rho_{c.s.} = q(1 + m) \tag{10}$$

In practice the density $\rho_{c.s.}$ of the slurry is determined by means of an areometer.

4. The total mass of cement is

$$G_c = k_c q V_{c.s.} \tag{11}$$

where k_c is a coefficient accounting for the losses of cement in loading-unloading operations, which is usually equal to 1.03-1.05.

The number n_1 of cement mixers required is determined proceeding from the calculated volume of the cement slurry on condition that each machine can be loaded with approximately 20 tons of cement:

$$n_1 = G_c/20$$

In this case the total volume of water required will be

$$V_w = mG_c/(k_c k_w \rho_w) \tag{12}$$

where k_w is a coefficient accounting for losses of water, equal to 1.03-1.05.

5. From Fig. 41 it follows that the volume $V_{d.\,f.}$ of the displacing fluid required to transport the cement slurry into the annular space is

$$V_{d.\,f.} = \Delta (\pi d_1^2/4)(L - h) \tag{13}$$

where Δ is the compressibility ratio of the displacing fluid, which varies within broad limits (from 1.01 to 1.10); L is the depth of well, m.

If the casing string is tapered, i. e., consists of several sections of different diameters, the required amount of the displacing fluid is then calculated by the following formula:

$$V_{d.\,f.} = \Delta \left[\sum_{i=1}^{i=n} (\pi d_i^2/4)\, l_i - (\pi d^2/4)\, h \right] \tag{14}$$

where n is the number of casing string sections differing in inside diameter; d_i is the inside diameters of casing string sections, m; l_i is the lengths of casing string sections, m; $\sum_{i=1}^{i=n} l_i$ is the landing depth of the casing string (approximately equal to the well depth L), m.

Usually the compressibility ratio Δ is taken at 1.03-1.05, but when making calculations for gas wells, the use of these values often results in that the calculated amount of the displacing fluid is 8-10 % less than that actually required, because of inaccurate calculation and measurement of the amount of the displacing fluid and a higher compressibility of the fluid (clay mud) due to the saturation of the fluid with gas.

Clay muds differ in their capability of being degassed. To calculate more accurately the amount of the displacing fluid required for cementing, it is necessary to determine the amount of gas (air) contained in the saturated fluid. This may be done by greatly diluting the fluid with water (in a proportion of 1 : 2 or 1 : 3), for with a sharp reduction of the viscosity and gel strength of gas-saturated clay mud, the gas freely separates from mud and the latter in a short period of time becomes practically completely degassed.

The proportion x of gas in the mud by volume is found from the formula

$$x = 3 - (\rho_{g.\,s.} + 2)/\rho_{d.\,m.} \tag{15}$$

where $\rho_{g.\,s.}$ and $\rho_{d.\,m.}$ are the densities of the gas-saturated clay mud and diluted clay mud (2 litres of water per 1 litre of clay mud), respectively.

For example, a gas-saturated clay mud with a density $\rho_{g.\,s.} = 1.3$ g/cm^3 is diluted with water in the ratio 1 : 2. The density $\rho_{d.\,m.}$ of the diluted mud has been found to be 1.12 g/cm^3, Consequently, under normal conditions the mud contains 5 % of gas, which amounts to 0.05 m^3 of gas per 1 m^3 of the mud. Knowing this relationship, we can determine the required amount of the displacing fluid, with due regard for its compressibility. The amount V of the displacing fluid is found by adding to the calculated amount $V_{d.\,f.}$ of the fluid the quantity $\Delta V_{d.\,f.}$ equal to the volume of gas contained in the fluid at a given proportion a of the gas by volume:

$$V = V_{d.\,f.} + aV_{d.\,f.} = V_{d.\,f.}(1 + a) \qquad (16)$$

i. e., $\Delta = 1 + a$.

6. The permissible pressure at the end of the displacement of the cement slurry may be determined from the formula

$$p = p_1 + p_2 \qquad (17)$$

where p_1 is the pressure required to overcome the difference in specific weight between the fluids in the casing and in the annular space

$$p_1 = \frac{1}{10}[(H - h)(\rho_{c.\,s.} - \rho_{d.\,f.})] \qquad (18)$$

p_2 is the pressure loss.

The pressure loss p_2 cannot as yet be accurately calculated. Experimental formulas are also not very accurate.

In approximate calculations use is sometimes made of the following empirical formulas.

(1) After N. Shatsov:

(a) for wells down to 1000 m deep

$$p_2 = 0.01L + 0.8 \text{ MPa}$$

(b) for wells more than 1000 m deep

$$p_2 = 0.01L + 1.6 \text{ MPa}$$

(2) After M. Krasin:

$$p_2 = 0.021L + 0.6 \text{ MPa}$$

(3) After V. Volovik:

$$p_2 = 0.001L \text{ MPa}$$

(4) After G. Gevinyan:

$$p_2 = \alpha B^{0.33} Q^{0.5} (1/\eta) L \text{ MPa}$$

where α is the coefficient dependent upon the diameter of the string and the delivery of the pump (0.10-0.20); B is the viscosity of the displacing fluid (clay mud) as measured with the СПВ-5 model viscometer, seconds; L is the depth of the well, thousand metres; η is the clear-opening factor of the well.

The results obtained by the above formulas considerably differ from one another.

The pressure developed by cementing units must be

$$p_{total} > p = p_1 + p_2$$

7. When calculating the permissible cementing time, it is necessary to provide for a time margin of 5-10 min per each 1000 metres of the well depth. The permissible cementing time T_{per} (in minutes) must correspond to the cement setting time with due regard for this time margin:

$$T_{per} \leqslant T_{set} + (5 \text{ to } 10)G \tag{19}$$

where C is the depth of the well in kilometres. The time margin in brackets is taken at 5 min for wells down to 2000 metres deep and at 8-10 min for deeper wells.

8. The required number of cementing units is

$$n = T_{tot}/T_{per} \tag{20}$$

where T_{tot} is the $t_p + t_d + 10$ min is the total time required to cement the well with one cementing unit; t_p is the time of pumping the cement slurry; t_d is the time of displacing the cement slurry; 10 min is the time required for releasing the cementing plug.

Under normal cementing conditions, practically all of the cement slurry (except when cementing deep wells where the slurry is to be raised to a great height) is pumped with the cementers operating in IV gear, i. e., at their maximum delivery. In this case, the total pressure loss p_2^{IV} must not exceed the maximum pressure $p_{c.u.}^{IV}$ developed by the cementing units:

$$p_2^{IV} \leqslant p_{c.u.}^{IV}$$

The time of pumping the cement slurry with the cementing units operating in IV gear then will be

$$t_p = V_{c.s.}/q^{IV} \tag{21}$$

where q^{IV} is the delivery of the cementing units when operating in IV gear.

After all of the cement slurry has been pumped down the well and the cementing plug released, the displacement of the slurry is started at the maximum possible rate in order to provide for the turbulent flow of the slurry in the annular space of the well.

If there is a danger of hydraulic fracturing of formations, the fluid pressure in displacing the slurry is limited by the fracturing pressure of the formations, and the displacement rate is then established on the basis of this pressure, or a cement slurry of lower density is specially selected.

Usually the displacement of the slurry starts with the cementing units running in IV gear. As the height of the slurry column in the annular space increases, the fluid pressure goes up, which requires shifting the cementing units into lower gears. As a rule, the process of cementing wells, especially of deep ones, ends with the cementing units running in I gear. The time of displacing the cement slurry may be found from the following formula:

$$t_d = \frac{V^{IV}_{d.f.}}{q^{IV}} + \frac{V^{III}_{d.f.}}{q^{III}} + \frac{V^{II}_{d.f.}}{q^{II}} + \frac{V^{I}_{d.f.}}{q^{I}} \qquad (22)$$

where $v^{IV}_{f.d.} \ldots v^{I}_{d.f.}$ are the amounts of the displacing fluid to be pumped into the well by the cementing units operating in the corresponding gears; $q^{IV} \ldots q^{I}$ are the deliveries of the cementing units when operating in the corresponding gears.

To determine the time t_d required for the displacement of the cement slurry, let us fix some position of the slurry in the well (Fig. 42) and write down a system of equations expressing the equality of the pressures in the casing and in the annular space, and the volume of the slurry in the casing and the annular space:

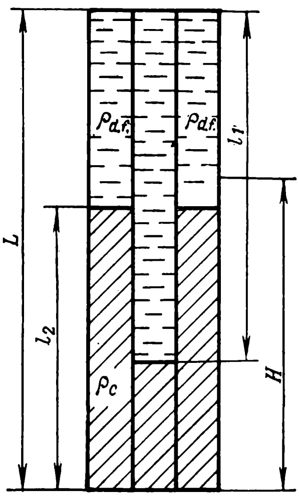

Fig. 42. Cementing process calculation diagram

$$(L - l_2)\rho_{d.f.} + l_2\rho_{c.s.} + 10p_2 = l_1\rho_{d.f.} + (L - l_1)\rho_{c.s.} + 10p_{c.u.} \qquad (23)$$

$$V_{c.s.} = \frac{\pi}{4}(D^2 - d^2)\,l_2 + \frac{\pi d_1^2}{4}(L - l_1) \qquad (24)$$

where $p_{c.u.}$ is the maximum pressure developed by the cementing units when operating in the nth gear; $V_{c.s.}$ is taken without considering the cavernosity ratio k.

Solving the system of Eqs. (23) and (24) for l_1 and denoting $\pi d_1^2/4 = F_1$ and $\pi(D^2 - d^2)/4 = F_2$, we get

$$l_1 = \frac{F_2L}{F_1 + F_2} + \frac{10F_2(p_{c.u.} - p_2)}{(F_1 + F_2)(\rho_{c.s.} + \rho_{d.f.})} + \frac{LF_1 - V_{c.s.}}{F_1 + F_2} \qquad (25)$$

where l_1 is the height of the displacing fluid column pumped into the casing at the maximum rate.

The height of the displacing fluid column in the casing when shifting the cementing units into lower gears is found in a similar way, but in this case the value of $p_{c.u.}$ for IV gear must be replaced by that of $p_{c.u.}$ for the given lower gear and l_1^{IV}, by l_1^{n}, where n = III, II, I (gears).

The total delivery of the cementing unit pumps must be such as to make the cement slurry flow through the annular space at a preset velocity ensuring complete displacement of the drilling mud:

$$Q_{c.\,u.} = 0.1\,(\pi/4)\left(D_m^2 - d^2\right) v_a \tag{26}$$

where v_a is the preset annular velocity of the cement slurry, m/s; D_m is the actual mean well bore diameter, m; d is the casing string outside diameter, m.

The total hydraulic power of the cementing units used must be sufficient for the units to develop, at a preset pumping rate, the necessary pressure to lift the cement slurry to a preset height:

$$N_{total} = 0.01\left(D_m^2 - d^2\right) p_{c.\,u.} v_a \tag{27}$$

where $p_{c.\,u.}$ is a preset pressure in the delivery line of the cementing units, MPa.

Proceeding from the data obtained on the basis of the above relationships and also from the technical data on the available cementing outfit, one can find the number of cementing units and cement mixers necessary for the successful conduct of the cementing process, i. e., for lifting the cement slurry to the preset height during the time allocated for this operation.

Thus, the necessary number of cementing units may be found from the formula

$$n_{c.\,u.} = N_{total}/N_{c.\,u.} \tag{28}$$

or

$$n_{c.\,u.} = Q_{c.\,u.}/q_{c.\,u.} \tag{28'}$$

where $N_{c.\,u.}$ is the hydraulic power of a single cementing unit, kW; $q_{c.\,u.}$ is the delivery of a single cementing unit, l/s.

If a fractional number is obtained, it should be rounded off to the nearest larger whole number. Also, provision should be made for one or two stand-by units.

The number of cement mixers participating simultaneously in the process of preparing the cement slurry is found proceeding from the condition that the rate of preparing the cement slurry should be the same as that of pumping the slurry into the well:

$$n_{c.\,m.} = Q_{c.\,u.}/q_{c.\,m.} \tag{29}$$

where $q_{c.\,m.}$ is the average delivery of a single cement mixer, l/s.

The total number of cement mixers required is determined with due regard for the necessary amount of dry cement:

$$n'_{c.\,m.} = G_c/g_{c.\,m.} \tag{30}$$

where $g_{c.\,m.}$ is the amount of dry cement loaded into the hopper of a single cement mixer under stationary conditions, t.

Hydraulic Cementing Process Calculations

Hydraulic casing cementing process calculations (in the given variant) are made with a view to determining the total delivery of cementing units that is necessary to ensure the maximum possible ascending drilling mud and cement slurry flow velocity in the annulus (i. e., the annular velocity), v_a at the given maximum permissible cementing-head pressure p_{ch} and bottom-hole pressure (or the maximum permissible pressure in the formation interval of the least fracture gradient), p_{bh}, and also selecting the necessary cementing equipment and determining the cementing time T_c to suit the design cementing process conditions and the cement thickening time.

The following boundary conditions are established:

$$p_{ch} \leqslant p_{wh}/1.5 \tag{31}$$

$$p_{bh} \leqslant p_{h.f.}/(1.2 \text{ to } 1.5) \tag{32}$$

$$T_c = t_p + t_d + 10 \leqslant 0.75 t_{thick} \tag{33}$$

where p_{wh} is the maximum permissible pressure on the wellhead equipment (cementing head, piping, pumps, etc.), MPa; $p_{h.f.}$ is the hydraulic fracturing pressure at the bottom of the hole or in the formation interval of the least fracture gradient, MPa; t_p is the cement slurry pumping time, h; t_d is the cement slurry displacement time, h; t_{thick} is the cement thickening time, as measured with a consistometer, h.

The following calculation sequence is recommended:

1. Proceeding from the actual geological and technical conditions in the given area and the adopted cementing practices, specify the maximum permissible annular velocity v_a by the end of cement slurry displacement when the danger of hydraulic fracturing of formations becomes the greatest.

2. The expected maximum cementing-head pressure at the specified v_a will be

$$p_{ch} = p_{hs} + p_c + p_a$$

where p_{hs} is the expected maximum difference in hydrostatic pressure between the casing and the annulus at the end of cementing; p_c is the pressure loss in the casing; p_a is the pressure loss in the annulus.

The magnitude of p_{hs} is calculated by the formula

$$p_{hs} = 0.1\,[(L - H)(\rho_{d.m.} - \rho_{d.f.}) + (H - h)(\rho_{c.s.} - \rho_{d.f.})]$$

where L is the well depth (casing string length); H is the height of cement slurry rise from casing shoe; h is the height of the cement plug in the string; $\rho_{d.m.}$, $\rho_{c.s.}$, and $\rho_{d.f.}$ are the respective densities of the drilling mud, cement slurry, and displacing fluid.

The magnitudes of p_c and p_a are found by the following formulas:

$$p_c = 82.6\lambda\rho_{c.\,s.}(Q^2L/d_{in}^5)$$

$$p_a = 82.6\lambda\rho_{c.\,s.}[Q^2L/(D-d_{out})^3(D+d_{out})]$$

$$Q = 0.785(D^2-d_{out}^2)v_a$$

where λ is the power loss factor taken at 0.035 for practical purposes; D is the weighted mean well bore diameter; d_{in} and d_{out} are the inside and outside casing diameters, respectively.

3. The expected maximum bottom-hole pressure will be

$$p_{bh} = p'_{hs} + p_a$$

where

$$p'_{hs} = 0.1\left[(L-H)\rho_{d.\,m.} + H\rho_{c.\,s.}\right]$$

4. Check the calculated magnitudes of p_{ch} and p_{bh} against the boundary conditions (31) and (32). If either of the conditions is not satisfied, correct the magnitude of v_a and repeat the calculations until both conditions are met.

5. Select the cementing unit model to suit the calculated values of Q and p_{ch} and determine the required number of such units from the relation

$$n_1 = Q/q$$

where q is the delivery of a single unit at a pressure $p \leqslant p_{ch}$.

6. It is recommended that the required number of cement mixers, n_2, should be determined from one of the following two relations, depending on the relationship between the calculated volume $V_{c.\,s.}$ of cement slurry and the inside volume $V_{in.\,c.}$ of the casing string to be cemented:

(a) if $V_{c.\,s.} \geqslant V_{in.\,c.}$, then $n_2 = Q/q_{c.\,m.}$, where $Q = n_1q$ is the total delivery of cementing units taking part in the pumping of the slurry, l/s, and $q_{c.\,m.}$ is the delivery of a single cement mixer, l/s;

(b) if $V_{c.\,s.} < V_{in.\,c.}$, $n_2 = G/G_{c.\,m.}$, where G is the total mass of dry cement required, t, and $G_{c.\,m.}$ is the hopper capacity (with reference to bulk mass) of a single cement mixer, t.

7. Determine the cementing time T_c to satisfy the boundary condition (33).

The prepared cement slurry is, as a rule pumped into the casing string at the maximum delivery of the cementing units. In this case,

$$t_p = 1000V_{c.\,s.}/60\Sigma q$$

where Σq is the total delivery of all cementing units that simultaneously take part in the pumping of the cement slurry.

Table 12

Cross-Sectional Area of Annulus Between Two Casing Strings as a Function of Relationship Between Their Diameters

Casing diameter, mm		Annulus area, m^2	Casing diameter, mm		Annulus area, m^2
OD	ID		OD	ID	
426	351	0.033	299	168	0.039
426	340	0.0385	299	146	0.0444
426	324	0.0471	273	219	0.0127
426	299	0.0594	273	194	0.0207
407	340	0.0259	273	178	0.0254
407	324	0.0346	273	168	0.0282
407	299	0.0468	273	146	0.0336
407	273	0.0585	273	140	0.0349
377	299	0.0295	273	127	0.0375
377	273	0.0412	273	114	0.0401
377	245	0.0526	245	194	0.0102
377	219	0.0621	245	178	0.0149
351	273	0.0271	245	168	0.0176
351	245	0.0385	245	146	0.0230
351	219	0.0480	245	140	0.0244
351	194	0.0560	245	127	0.0268
340	245	0.0330	245	114	0.0295
340	219	0.0425	219	168	0.00895
340	194	0.0505	219	146	0.0142
340	178	0.0552	219	140	0.0157
324	245	0.0254	219	127	0.0186
324	219	0.0350	219	114	0.0209
324	194	0.0430	194	146	0.00652
324	178	0.0476	194	140	0.00785
324	168	0.0504	194	127	0.0104
299	245	0.0140	194	114	0.0130
299	219	0.0235	178	127	0.0073
299	194	0.0316	178	114	0.0099
299	178	0.0362	168	114	0.00745

Where $V_{c.\,s.} > V_{in.\,c.}$, the last portions of cement slurry are pumped at a slower rate, and so the pumping time t_p should be calculated accordingly.

The displacement of the cement slurry is usually started at the maximum delivery of the cementing units. As the slurry flows through the annular space and the height of its column in the annulus increases, the cementing-head pressure gradually grows too high, so that the pumping rate has to be progressively reduced at certain moments.

When specifying the delivery of the cementing units participating in the pumping and displacing of the cement slurry, one should proceed from the maximum annular velocity that can safely be permitted without running the risk of hydraulic fracturing of formations.

The total cement slurry displacement time is given by the expression

$$t_d = \Sigma V_{d.\,f.\,i}/Q_i$$

where $V_{d.\,f.\,i}$ is the displacing fluid volume pumped into the well with the cementing units operating in the ith gear; Q_i is the total delivery of the cementing units in the ith gear.

Table 13

Theoretical Volume (in m³) of 1 m of Inside Space of Casing Pipes as a Function of Their Diameter and Wall Thickness

Casing pipe diameter, mm	Casing pipe wall thickness, mm							
	6	6.5	7	8	9	10	11	12
114	0.0082	—	0.0078	0.0075	0.0072	—	—	—
127	0.0103	—	0.0101	0.0097	0.0093	—	—	—
140	0.0131	—	0.0127	0.0123	0.0119	0.0115	0.0112	—
146	—	0.0141	0.0137	0.0133	0.0129	0.0125	0.0121	—
168	—	0.0191	0.0186	0.0181	0.0177	0.0172	0.0167	0.0163
178	—	—	0.0211	0.0206	0.0201	0.0196	0.0192	0.0186
194	—	—	0.0254	0.0249	0.0243	0.0238	—	0.0227
219	—	—	0.0330	0.0323	0.0317	0.0311	—	0.0298
245	—	—	0.0419	0.0412	0.0405	0.0397	—	0.0383
273	—	—	0.0526	0.0518	0.0510	0.0502	—	0.0495
299	—	—	—	0.0628	0.0620	0.0611	0.0602	0.0593
324	—	—	—	—	0.0736	0.0730	0.0719	0.0711
340	—	—	—	—	0.0814	0.0803	0.0795	0.0786
351	—	—	—	—	0.0870	0.0860	0.0849	0.0839
377	—	—	—	—	0.1020	0.1000	0.0989	0.0978
407	—	—	—	—	0.1190	0.1170	0.1160	0.1150
426	—	—	—	—	—	0.1290	0.1280	0.1270
508	—	—	—	—	—	—	0.1850	—

The magnitude of $V_{d.\,f.\,i}$ is found by solving a system of equations set up in accordance with the diagram of Fig. 42:

$$(L - l_2)\,\rho_{d.f.} + l_2\rho_{c.\,s.} + 10\,(p_c + p_a) = l_1\rho_{d.\,f.} + (L - l_1)\,\rho_{c.\,s.} + 10p_{c.\,u.\,i}$$

$$V_{c.s.} = 0.785\left(D^2 - d_{out}^2\right)l_2 + 0.785d_{in}^2\left(L - l_1\right)$$

where $p_{c.\,u.\,i}$ is the maximum pressure developed by the cementing units in the ith gear; l_1 and l_2 are the respective cement slurry levels in the casing and the annular space by the end of cementing unit operation in the ith gear.

Denoting $F_1 = 0.785\,d_{in}^2$ and $F_2 = 0.785\left(D^2 - d_{out}^2\right)$ and solving the above system of equations for l_1, we get

$$l_1 = \frac{F_2L}{F_1 + F_2} + \frac{10F_2\left(p_{c.\,u.\,i} - p_c - p_a\right)}{\left(F_1 + F_2\right)\left(\rho_{c.\,s.} - \rho_{d.\,f.}\right)} - \frac{LF_1 - V_{c.\,s.}}{F_1 + F_2}$$

Table 14

Cross-Sectional Area of Annulus Between Well Walls and Casing String as a Function of Well Bore and Casing Pipe Diameters (without regard to cavernosity ratio)

Diameter, mm		Annulus area, m²	Diameter, mm		Annulus area, m²
well bore	casing pipe		well bore	casing pipe	
540	426	0.0864	269	219	0.0192
490	426	0.0460	320	194	0.0509
490	407	0.0589	295	194	0.0388
445	407	0.0259	269	194	0.0273
490	377	0.0769	243	194	0.0168
445	377	0.0438	295	178	0.0443
490	351	0.0918	269	178	0.0318
445	351	0.0587	243	178	0.0214
394	351	0.0251	214	178	0.0111
445	340	0.0644	295	168	0.0461
394	340	0.0306	269	168	0.0346
370	340	0.0165	243	168	0.0242
445	324	0.0725	214	168	0.0138
394	324	0.0390	190	168	0.0062
370	324	0.0251	269	146	0.0401
445	299	0.0752	243	146	0.0297
394	299	0.0517	214	146	0.0192
370	299	0.0373	190	146	0.0116
346	299	0.0232	243	140	0.0308
394	273	0.0634	214	140	0.0203
370	273	0.0490	190	140	0.0127
346	273	0.0355	161	140	0.0050
320	273	0.0216	243	127	0.0337
370	245	0.0604	214	127	0.0232
346	245	0.0469	190	127	0.0156
320	245	0.0333	161	127	0.0075
295	245	0.0212	214	114	0.0258
346	219	0.0564	190	114	0.0181
320	219	0.0428	161	114	0.0101
295	219	0.0307	145	114	0.0063

Using the obtained values of l_1 for each working speed of the cementing units, determine the corresponding magnitudes of $V_{d.f.i}$ and then find t_d.

8. Select the composition of the cement slurry so as to have t_{thick} satisfy the boundary condition (33).

The above calculations can be made easier by using the reference data listed in Tables 12 through 15.

Practical Studies

Calculate a full-cover (continuous) single-stage casing cementing process for the following case: well depth $L = 2500$ m, cementing interval length $l_2 = 2000$ m, cement plug height

Table 15

Theoretical Well Bore Volume (in m^3) as a Function of Well Depth and Bit Diameter

Well depth, m	Drill bit diameter, mm											
	190	214	243	269	295	320	346	370	394	445	490	540
50	1.42	1.80	2.32	2.84	3.42	4.02	4.70	5.38	6.10	7.77	9.43	11.45
100	2.83	3.60	4.64	5.68	6.83	8.04	9.40	10.75	12.19	15.54	18.85	22.89
200	5.67	7.20	9.27	11.36	13.66	16.08	18.80	21.50	24.38	31.08	37.70	45.78
300	8.50	10.80	13.91	17.04	20.49	24.11	28.19	32.25	36.57	46.62	56.55	68.67
400	11.33	14.38	18.54	22.72	27.32	32.15	37.59	43.00	48.76	62.16	75.40	91.56
500	14.17	17.98	23.18	28.40	34.17	40.19	46.99	53.75	60.95	77.70	94.25	114.50
600	17.00	21.57	27.81	34.08	40.99	48.23	56.39	64.50	73.14	93.24	113.10	137.30
700	19.83	25.17	32.45	39.76	47.82	56.27	65.79	75.25	85.33	108.80	132.00	160.20
800	22.66	28.76	37.08	45.44	54.65	64.30	75.18	86.00	97.52	124.30	150.80	183.10
900	25.50	32.36	41.72	51.12	61.49	72.34	84.58	96.75	109.70	139.90	169.70	206.00
1000	28.33	35.95	46.35	56.80	68.31	80.38	93.98	107.50	121.90	155.40	188.50	228.90
2000	56.66	71.90	92.70	113.60	136.60	160.80	188.00	215.00	243.80	310.80	377.00	457.80
3000	84.99	107.80	139.10	170.40	204.90	241.10	281.90	322.50	365.70	466.20	565.50	676.70
4000	113.30	143.80	185.40	227.20	273.20	321.50	375.90	430.00	487.60	621.60	754.00	905.60

$h = 20$ m, well bore (drill bit) diameter $D = 243$ mm, casing outside diameter $d_{out} = 146$ mm, casing inside diameter $d_{in} = 130$ mm, displacing fluid (drilling mud) density $\rho_{d.\,f.} = 1.2$ g/cm^3, cement slurry density $\rho_{c.\,s.} = 1.9$ g/cm^3, cavernosity ratio $k = 1.3$.

Laboratory Work No. 1

Determination of the compressibility ratio Δ of a gas-saturated clay mud.

1. Take 1 liter of the test clay mud of density $\rho_{g.\,s.}$ and dilute it with water in the ratio 1 : 2 to obtain a mixture of density $\rho_{d.\,m.}$.
2. Agitate the mixture in a vessel until gas bubbles stop escaping from the mixture.
3. Using the formula $x = 3 - (\rho_{g.\,s.} + 2)/\rho_{d.\,m.}$, calculate the proportion (by volume) of the gas in the mud and then determine Δ.

Chapter 4

Cementing Conditions and Requirements for the Quality of Cement Slurries and Stone

Requirements on plugging materials for cementing oil and gas wells are determined by geological and technical conditions existing in the wells. The problem of choosing proper materials is a complex one. The plugging mixture (slurry) should remain mobile during its transport into the annular space and, right after the termination of the process of cementation, must harden into a stone exhibiting definite physical and mechanical properties. These processes take place in the well bore where the temperature and pressure vary with depth and where there exist high-pressure and intake beds, and also beds carrying mineralized waters, petroleum, and gas. Under such varying conditions, a single type of cement or one and the same composition of the plugging mixture (slurry) cannot be equally acceptable. One type of cement cannot possibly satisfy all the requirements stemming from the variety of conditions prevailing even in a single well.

Future prospects for super-deep drilling have at present acquired such dimensions that a number of areas have already been singled out for sinking wells to a depth down to 15 000 metres.

4.1. Temperature and Pressure in Wells

Among the major factors determining the choice of plugging materials, especially in the case of deep and super-deep wells, are the temperature, pressure and the composition of mineralized stratal waters.

An increase in the depth of oil and gas wells is attended by a continual rise of the bottom-hole temperatures. In the areas drilled over in the Soviet Union, the bottom-hole temperatures vary within a very wide range.

Within the intervals marking the occurrence of prevalently terrigenous tertiary and, partially, cretaceous deposits in the Krasnodar Territory mean geothermal gradients * down to a depth of approximately 2700 m vary in the range from 2.25 to 2.59 °C/100 m. By 1970, depths of 4500-6000 m were reached, where the temperature of the surrounding rocks is as high as 190-200 °C.

In the tertiary and cretaceous deposits of the oil- and gas-bearing districts of the Stavropol Territory, the values of geothermal gradient down to a depth of 2100 m lie in the range from 3.48 to 8.61 °C/100 m. In the Maikop deposits, the geothermal gradient reaches as high a figure as 10.7 °C/100 m. In the areas of Ozek-Suat, Zimnyaya Stavka, Kamysh-Burun, the temperature at depths of about 4000 m reaches 140-150 °C. Bottom-hole temperatures of 175-186° C are recorded in the wells 6 Praskoveiskaya, 1, 2 Aleksandrovskaya and 18 Zhuravskaya. In the well 1 Galyugaevskaya the temperature at a depth of 5500 m amounted to about 200 °C. In the areas of the Krasnodar Territory, the highest temperature was recorded in the well 1 Medvedovskaya, where it stood at 216 °C at a depth of 6087 m.

In Chechen-Ingushetia, the geothermal characteristics of drill logs are distinguished by a considerable influence exercised by the circulation of stratal waters, this giving rise to the emergence of heat centres at the sites marking the occurrence of aquifers. Because of this, the geothermal gradient varies along the vertical section within a significantly wide range and in deposits overlapping water-bearing horizons it reaches 12-16 °C/100 m. While drilling wells in the Uzbek SSR, a temperature of 224 °C was registered at a depth of 5500 m.

In the practice of drilling, a temperature of 237.7 °C was noted (South-Western Texas, USA).

In choosing the composition of a plugging mixture for cementing wells, it is not always possible to accurately estimate from the established geothermal gradient the actual bottom-hole temperatures.

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* The term "geothermal gradient" means the rate of increase of temperature downward in the Earth. The unit of measurement of the geothermal gradient is °C/100 m.

High temperatures have a marked effect not only on the technology of drilling, casing, and cementing wells, but also on the choice of materials used. Increasing well depths to 10 000-15 000 m will still more increase the role of the temperature factor in the processes involved in the drilling of wells. Therefore, exact evaluation of bottom-hole temperatures in super-deep wells is of paramount importance.

On the surface of the Earth, temperature fluctuates as a function of the time of the day and season of the year. The amplitude of surface temperature variations rapidly decreases with depth. The thermal waves produced by diurnal temperature fluctuations penetrate to a depth of 1 metre. The penetration depth of annual waves is much greater, reaching 30 metres and more.

The depth below which the effect of surface temperature is virtually absent is called the depth of the "neutral layer". In different regions this depth varies from 15 to 30 m. Below the neutral layer, temperature continuously rises with depth.

Operative in the earth shell are rock pressure, which characterizes the naturally stressed state of the rocks, and the pressure of fluids that are contained in the rocks. These pressures determine the needed parameters of flushing fluids and the forces acting on casing strings and bottom-hole and wellhead equipments.

The rock pressure, as the stress existing in rocks, is due to the action of the forces of gravity from the superjacent rock layers, tectonic processes, thermal field variations, physico-chemical processes of formation and transformations of rocks, etc. A distinction is made between the vertical and horizontal components of the rock pressure, which are termed total and lateral rock pressure, respectively.

Usually, the fluid pressure in deposits of water, oil, and gas approximately equals the so-called conventional hydrostatic pressure which is defined as the pressure of a column of sweet water whose height is equal to the cover thickness of the deposit. But in existence are deposits where fluid pressures exceed the ordinary values by a factor of 1.3-1.6 and even reach the level of the rock pressure. Such pressures are called abnormally high formation pressures (AHFP).

The number of oil and gas pools with AHFP at depths of 3500 to 4000 m is comparatively insignificant and does not exceed 10 %-15 %. With further increase in the occurrence depth, the percentage of fluid accumulations featuring AHFP grows higher, and the deeper such an accumulation occurs, the greater the absolute value of AHFP.

The formation pressure may also be below the hydrostatic one.

The ratio of the formation pressure to the water column pressure at a given depth is given the name of abnormality factor:

$$a = p_f / p_w$$

In the USSR, there are beds with an abnormality factor of 1.5 to 2.0 and higher (Krasnodar Territory, Chechen-Ingush and Dagestan Autonomous SSR, Ukrainian, Uzbek, Azerbaijan SSR and other regions).

In cementing wells, one should know both static and dynamic temperatures. The static temperature is the one prevailing in rocks of an intact block. In wells, the bottom-hole temperature is taken to be approximately equal to the static temperature, provided that the circulation of the drilling mud through them has been stopped for 2 to 4 days.

By the dynamic temperature is meant a steady (constant) temperature at a certain depth in the well during circulation of the drilling mud therein. In practice, it is considered that constant dynamic temperature sets in after one or two cycles of the drilling mud circulation. The dynamic temperature always stands below the static one. The difference between these temperatures depends upon a number of geological, technical and technological conditions and in wells up to 6000 m deep, it ranges from 10 to 40 °C. This difference, however, should be verified in each concrete instance.

The composition of a plugging mixture for primary cementing is chosen by taking into consideration the dynamic temperature. In recementing jobs, plugging mixtures are selected on the grounds of the static temperature.

It is with regard to the joint action of temperature and pressure that the composition of plugging mixtures should be chosen and their physico-mechanical properties determined.

4.2. Stratal Waters

Mineralized waters contained in formations may shorten the setting time of a plugging mixture moving in the annulus of a well. A combined action of temperature, pressure, and stratal waters destroys the stone formed by many types of plugging cements. For this reason, the choice of the type of cement, with consideration for its resistance to attack by mineralized stratal waters, should be given serious attention.

According to the classification proposed by G. M. Sukharev, the waters of oil and gas pools may be subdivided into sodium sulphate, sodium hydrocarbonate, magnesium chloride and calcium chloride ones. Most prevalent in oil deposits are sodium hydrocarbonate waters. Sodium hydrocarbonate waters occurring in the oil fields of the USSR are frequently found to belong to the subgroups of chloride-alkali waters.

Gases contained in subterranian waters have a pronounced effect on their corrosion activity. Waters in zones of difficult water exchange most frequently contain hydrocarbon gases, carbon dioxide, and hydrogen sulphide. The reactivity of stratal waters increases with rising temperature.

The data presented in Table 16 may serve as an example illustrating the composition of mineralized stratal waters.

Table 16

Chemical Composition of Stratal Waters (deposits of the Krasnodar Territory)

Site of water sampling	Ion content, 10^{-3} mol					
	Cl^-	HCO_3^-	SO_4^{2-}	Ca^{2+}	Mg^{2+}	$Na^+ + K^+$
Well 1 Medvedovskaya, 4280-4265 m deep (Lower Cretaceous)	930.0	6.40	1.28	227.00	14.06	696.60
Well 6 Yuzhno-Sovetskaya, 3008-3003 m deep (Lower Cretaceous)	263.0	18.80	3.77	5.05	1.55	278.90
Well 26 Severo-Akhtarskaya, 1050-1029 m deep (Karagan)	779.7	20.54	0.96	14.65	3.67	782.91

Note. No CO_3^{2-} ions.

4.3. Requirements to the Quality of Plugging Mixtures and Stone

Mobility of plugging mixtures. The most important property of any plugging mixture is its mobility, i. e., the capability of being easily pumped by pipes during the entire period of time that is required to complete the cementing job. The mobility (fluidity) of a slurry is determined by means of a special cone (similar to the one used in the slump test of concrete) developed at the Azerbaijan Scientific Research Institute for Petroleum Production and must be at least 18 cm. This property of plugging mixtures depends on the nature of the binder used, the fineness of grind, water-cement ratio, the amount, impurity content, and specific surface of the aggregate (filler), and the applied additives, and also on the conditions under which the mixtures remain throughout the process of cementing and the time and method of their preparation (mixing).

The above method of determining the mobility of slurries enables one to adjust quickly the amount of water in the slurry of a given composition and is considered only as an approximate method for establishing the consistency of slurries. It is recommended that the fluidity of slurries for cementing deep wells with small casing clearances should be increased to 22 cm. According to the USSR Standard GOST 1581-78, the mobility of slurries must be such as to allow them to spread over a circle of at least 180 mm in diameter in the fluidity test at a water-cement ratio of 0.5.

The density of a plugging mixture is one of its main characteristics. In cementing of wells, the density of the plugging mixture used is practically the only criterion so far for evaluating its quality.

Variations in the density of the plugging mixture in the course of cementing point to changes in its water-cement ratio. Such fluctuations are held to be a factor upsetting the technological cycle of the process and may lead to complications, for instance, to a rise of pressure while cementing. It proves particularly difficult in practical work to keep to a specified formulation when mixing cement with water and other ingredients to yield lightened plugging mixtures. A decrease in the density of a slurry means an increase in its water-cement ratio, which impairs the properties of the stone formed by the slurry.

Bearing in mind that water-cement ratio determines also other physical and mechanical properties, a strict control over changes in the density of plugging mixtures is necessary in doing cementing jobs and no significant deviations from the pre-assigned values should be allowed. The cementing process usually proceeds normally when variations in the density of the plugging mixture do not exceed 0.02 gf/cm^3.

Setting time of plugging mixtures. The suitability of a plugging mixture for bringing it into the annular space of a well is appraised by its setting time. To determine the setting time of plugging mixtures at 22 and 75 °C a device called Vicat apparatus, or cement needle, is employed.

By the initial set is understood the time lapsed from the instant when cement is mixed with water till the moment when the needle, while sinking into the mixture, stops at a distance of 0.5 to 1.0 mm from the glass plate.

By the final set of a plugging mixture is meant the time passed from the moment at which the cement is mixed with water to that when the needle, dipping into the mixture, penetrates it down to a depth of 1 mm.

To determine the setting time of plugging mixtures at high temperatures and pressures, use is made of a special apparatus — an autoclave — designed to operate under a working pressure of up to 100 MPa and at a high temperature.

The autoclave (Fig. 43) consists of body *1*, bottom *2*, lock *3* with seal *4*, and holder *5* with Vicat needles *6*. The weight of each needle, when immersed in water, is 300 ± 1 g. The holder may accommodate from 6 to 12 needles, depending on the design of the autoclave. The body, lock, bottom, and seals of the autoclave are designed to withstand the specified maximum test pressures and temperatures.

Placed in the lower part of the holder is Vicat ring *15*. The needles are dropped by turning handle *7* held to shaft *8* that passes through lock *3* and is sealed in it by means of packing *9*. Shaft *8* carries on its lower end disc *10* from whose edge the Vicat needles are suspended. The disc has a segment-shaped recess whose position is indicated by pointer *12* moving over dial *11*. When the recess in the disc is brought in register with a needle, the latter is released and falls into the Vicat ring. As the autoclave is being pressurized, air is let out of it through hollow needle *13*. The sending unit of a temperature gauge is placed in pocket *14* filled with transformer oil.

The inside surface of the Vicat ring and the bottom plate upon which the ring is placed are oiled with machine oil prior to every test. The ring is then placed on the plate and filled with water to check the joint between the ring and the plate for leaks. If water leaks from under the ring, the latter should be rejected.

A sample of the plugging mixture to be tested is then poured in the Vicat ring, the surface of the mixture in the ring is smoothed out level with the ring edges with the aid of a knife blade, and the ring is installed in the holder already loaded with the needles. The holder is placed in the autoclave which is then filled with water (via connection *16*), locked, and pressurized. The initial pressure in the autoclave (2.5 to 5 MPa) is determined by the force with which the seal of the autoclave lock is compressed when pressurizing the autoclave. The start of

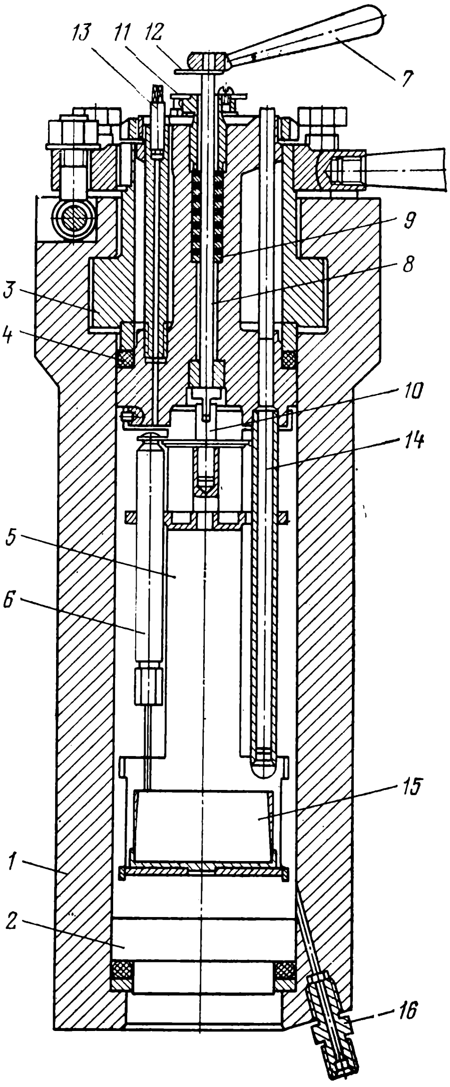

Fig. 43. Autoclave

the test is indicated on the temperature and pressure recorder charts. Indicated are the date, serial number, and starting time of the test.

If the temperature in the autoclave reaches the specified value while the pressure stays too low, use should be made of a hydraulic press to bring the pressure in the autoclave to the specified test value. Once the specified temperature and pressure values in the autoclave have been reached, the autoclave heater current should be reduced to a value at which thermal equilibrium sets in.

The first Vicat needle is dropped at the moment specified in the test assignment, whereas the moment to release the next needle is determined depending on the results of dropping the first one. If the first needle drops with a clear ringing knock, the mixture under test is still liquid. If the knock is dull or if there is no knock at all, the needles are dropped one every 15 minutes. After dropping the last needle, the heater is switched off and the autoclave is let to cool to 75 °C. Then the excess pressure in the autoclave is released, the sample is removed, and the initial and final setting times of the test mixture are determined by measuring the penetration depths of the needles into the sample. The results are entered in the test log.

Should it prove impossible to establish either of these times experimentally, the penetration depth of the needles is then plotted as a function of time and the initial and final setting times of the mixture under test are found by extrapolation. The test is repeated thrice and the setting times of the mixture are determined as the arithmetic mean of the three results.

The setting times of plugging mixtures are adjusted to suit concrete cementing conditions.

Consistency of plugging mixtures. In cementing deep, high-temperature wells, one must know not only the initial and final set of plugging mixtures under steady-state conditions, but also changes in their consistency (i. e., thickness) with time in conditions of constant agitation.

The consistency of plugging mixtures is measured with the aid of special apparatus called consistometers. The КЦ-3 Model consistometer is designed to operate at pressures up to 100 MPa and temperatures up to 200 °C, whereas the КЦ-4 Model, at pressures up to 150 MPa and temperatures from 250 to 300 °C. Both models are similar in construction. Their component parts and assemblies are mounted on a single frame in a common housing divided into two compartments, one of them accommodating the hydraulic system and the other, the electrical system of the apparatus.

A schematic diagram of the КЦ-3 Model consistometer is shown in Fig. 44. The operating principle of the apparatus is based on measuring the torque acting on paddle *17* when cup *18* containing the test mixture, in which the paddle is immersed, is rota-

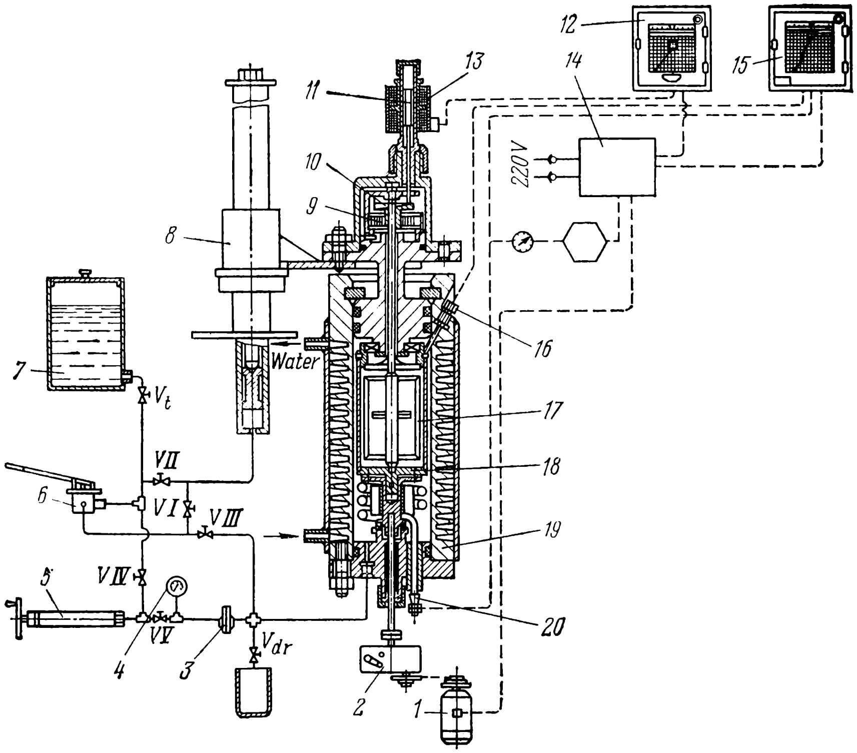

Fig. 44. Consistometer for determining the thickening time of plugging mixtures

ted with a specified speed. The paddle is kept from rotating by measuring spring *9* whose twist is converted by face-cam device *10* into the linear motion of plunger *11* of differential-transformer transducer *13* and recorded by the ДСМР-2 Model small-size recorder *12* supplied from power unit *14*. The body of transducer *13*, whose interior communicates with high-pressure autoclave *19*, and the rod of plunger *11* are made of a nonmagnetic material, which makes it possible to compensate for changes in their linear dimensions caused by temperature fluctuations.

Cup *18* filled with the slurry to be tested is placed, together with the paddle device, in the autoclave which is then filled with oil. The oil is separated from the slurry by means of a rubber diaphragm. Internal tubular electric heater *20* serves to heat the oil in the autoclave.

To ensure a uniform heating of the oil, the outside surface of cup *18* is provided with a helical groove which makes the oil flow downwards when the cup is rotating. To reduce dissipation of heat, the autoclave is provided with a heat-insulating shell. The heating rate of the apparatus is 3 to 5 °C per minute and can be controlled by varying the heater power. The temperature in the apparatus is monitored, recorded, and automatically controlled by means of control indicating and recording potentiometer *15* connected to thermocouple *16*.

The autoclave is filled with oil from tank *7* by means of hand-operated plunger pump *6*. The necessary pressure in the autoclave is built up and controlled with the aid of hand-operated hydraulic press *5*, designed to handle pressures up to 100 MPa, and booster *10*. To monitor the pressure, use is made of pressure gauge *4*. Trap (separator) *3* serves to transmit the pressure developed by the hydraulic press into the oil line leading to the autoclave.

Cup *18* receives rotation from electric motor *1* via two-speed gearbox *2*.

By passing water between the finned surface and the shell of the autoclave, the latter can be cooled from its maximum temperature to a temperature of 40 to 50 °C in 15 minutes.

The autoclave is easy and quick to open, thanks to its quick-release locking arrangement — a fork. The autoclave cover is lifted by means of hydraulic lifter *8* actuated by hand-operated plunger pump *6*.

The test procedure is quite simple. The slurry to be tested is poured in cup *18* which is then placed, together with the paddle held to the autoclave cover, into the autoclave. After pressurizing the apparatus, the temperature and pressure in it are raised to the specified values. The cup is set in rotation, which ensures continuous stirring of the slurry in the cup and makes the oil contained in the autoclave circulate. The consistency of the slurry is recorded as a function of time on the chart strip of recorder *15* that is calibrated to read viscosity in N s/m^2 (Pa s). The curve recorded on the chart strip features a sharp inflection followed by a steep rise and is used to determine what is known as the thickening, or pumpability, time of the slurry. The limiting value of viscosity for cement slurries, as recorded by the consistometer, is conventionally set at 10 Pa s, and the time it takes the slurry under test to reach this value is the thickening time. However, experience has shown it advisable to take the limiting value of viscosity for plugging mixtures at 5 Pa s, for in conditions of high temperatures and pressures the viscosity of these mixtures grows intensively. Once the test is completed, the apparatus is cooled and the sample is removed from it.

The КЦ-5 Model consistometer is designed to determine the thickening time of plugging mixtures at atmospheric pressure and temperatures of 90 to 95 °C. It is simpler in construction than the above-described consistometer models and is specially suited for testing cement slurries to be used in wells with mild downhole conditions.

Foaming. When pumping cement slurry into the well, one must provide not only for an accurate estimation of the volume of the slurry being pumped, but also for uninterrupted operation of the pumps.

The foaming of cement slurries upon their treatment with various chemicals, e. g., spent sulfite-alcohol liquor, is fraught with grave consequences. Where the slurry is treated with large amounts of spent sulfite-alcohol liquor, much foam is produced in the course of mixing, which greatly hampers the cementing job and, what is more, causes errors in estimating the volume of the slurry pumped into the well and its density.

For this reason, the foaming ability of any cement slurry must be determined beforehand under laboratory conditions.

Water (filter) loss of cement slurries. One of the most important properties of cement slurries is their sedimentation stability, i. e., the ability to stay for a long period of time without separating into liquid and solid phases. On standing, a slurry of inadequate sedimentation stability gets segregated into water and cement paste zones resulting in discontinuities in the cement stone formed in the annular space.

An effective means for improving the sedimentation stability of cement slurries is to reduce their water, or filter, loss.

Figure 45 shows a schematic diagram of the hydraulic part of the УВЦ-1 Model apparatus for measuring the water loss of cement slurries and permeability of cement stone.

The УВЦ-1 Model apparatus is divided into two individual parts — hydraulic and electrical — mounted on a common frame. The hydraulic part includes autoclave *1*, booster *2* with recorder *3*, hydraulic press *4*, hand-operated pump *5*, a safety valve, a manifold, two indicating and one recording pressure gauges, and a tank.

The electrical compartment of the apparatus houses the ПСМР-2 Model potentiometer, the ДСМР-2 Model secondary recording pressure gauge, and the ЛАТР Model variable-ratio laboratory autotransformer. Mounted on the face wall of the apparatus are an ammeter and a control panel.

Autoclave *1* consists of body *6*, insert *7*, and heat-insulating shell *8*. The insert is held in the autoclave body by means of special fork-shaped lock *9*.

Cut in the external cylindrical surface of the autoclave body is a helical groove (to provide for the passage of cooling water) closed on the outside by a metal jacket that is welded to the body.

Coiled tubular electric heater *10* and a thermocouple pocket are held fast in the bottom of the autoclave body. Inside the heater coil, there is removable cup *11* with stirrer *12* inserted into it, which is intended to hold the slurry under test. The stirrer is driven through a coupling from spindle *13* arranged in the center of insert *7*. The top end of spindle *13* carries a belt pulley to connect the spindle to a drive reduction gear. The upper part of

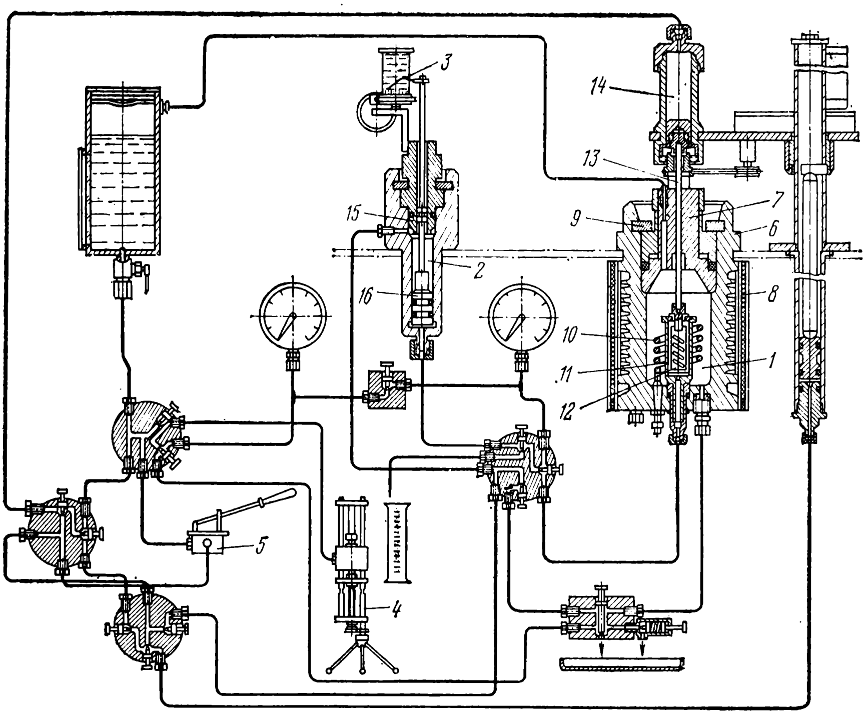

Fig. 45. УВЦ-1 Model apparatus for determining the water loss of plugging mixtures

insert *7* is attached to the piston of lifter *14* that allows the stirrer to be lifted from the cup containing the test slurry. The necessary pressure differentials for determining the water loss of slurries and permeability of cement stone are produced by booster *2* whose body accommodates changeable bushing *15* and piston *16*.

The water loss of plugging mixtures must be low, close to that of high-standard clay drilling muds.

In specifying the maximum permissible water loss of a cement slurry, one must bear in mind (1) the effect of water loss on the pumpability of the slurry in the course of cementing and (2) the effect of the ingress of the slurry filtrate into the producing formation on the properties of the latter as a collector.

As the slurry loses water through filtration, it gradually grows thick and eventually, with too much water filtered off, may lose its pumpability, which, as a rule, causes complications. The ingress of the slurry filtrate into the producing formation impairs its permeability, which tends to prolong the well completion period.

Mechanical strength of cement stone. The mechanical strength of cement (plugging) stone is, as yet, the principal estimative characteristic of plugging cements. The mechanical properties of cement stone are characterized by its bending strength (USSR), found by testing standard beam test pieces, or compressive strength (USA) in testing standard cylindrical test pieces.

According to the pertinent USSR standard, cement stone is to be tested for strength after two days' hardening in an aqueous medium at a certain (standard) temperature. The standard specifies the properties of cement stone as a material, but where a primary casing cementing job is concerned, both theoretical and experimental studies and field experience show that the requirements for the strength of cement stone may be alleviated.

In the cemented annulus of a well, there may develop tensile, compressive, and bending stresses, but the actual type of deformation occurring in the cemented annular space has so far been found to have no direct bearing on the choice of a particular method for testing cement stone specimens. Experience shows that any type of strength test may be adopted for evaluating the quality of plugging cements. Preference, however, is given to bending and compression tests. Compression tests are convenient to perform, and bending tests on standard beam test pieces allow one to subject the test-piece halves remaining after the bending tests to compression tests to obtain additional information.

Permeability of portland cement stone. A dense, impervious cement stone whose permeability does not increase under the effect of various factors (hardening conditions, stratal waters, etc.) is much more resistant to attack by corrosive waters, for it suffers no volumetric destruction, suffusion, leaching, etc. Casing pipes, usually liable to corrosion by stratal waters, serve longer if protected by such a stone. In certain conditions, the permeability of cement stone may be responsible for water influx into wells and oil and gas crossflow between beds. With waters or gas filtering through it, the stone disintegrates more intensively.

There are no standards for the permeability of cement stone. However, experience and appropriate calculations show that to effectively isolate producing horizons, use should be made of plugging mixtures that turn into stone with a permeability of $(2 \text{ to } 4) \times 10^{-3} \mu^2$.

Chapter 5

Composition and Basic Properties of Portland Cement

5.1. Classification of Plugging (Oil-Well) Cements and Mixtures

According to their binder base, plugging cements are classed in several types as follows: (a) plugging cements using portland cement as their base; (b) plugging cements based on blast-furnace slags; (c) belite-diatomaceous earth cements; (d) lime-sand cements; (e) other types of plugging cement (gypsum cements, cements with natural minerals and rocks as their hase); (f) organic binder-base cements.

As regards their test temperature and application field, plugging cements are divided into the following three classes.

I. Cements for "cold" wells, or low-temperature cements, whose test temperature is 22 ± 2 °C.

II. Cements for "hot" wells, or high-temperature cements, with a test temperature of 75 ± 3 C.

III. Cements for deep, high-temperature wells.

As to their mixing fluid, plugging mixtures may be classified into aqueous, aqueous-emulsion, and oil-cement (diesel fuel, kerosene, various types of petroleum, etc.) mixtures.

With respect to the additives — fillers (aggregates), oil-well cements are subdivided into sand, fibrous, gel-cement, pozzolan, perlite, hematite-magnetite, bentonite and others.

According to their density, plugging mixtures are classed as light (up to 1.3 g/cm^3 in density), lightened (from 1.3 to 1.75 g/cm^3 in density), normal (from 1.75 to 1.95 g/cm^3 in density), weighted (from 1.95 to 2.2 g/cm^3 in density), and heavy (over 2.2 g/cm^3 in density).

There exist plugging mixtures with ordinary (normal) and low water-loss properties.

With regard to their setting time, plugging mixtures may be subdivided into quick-set (initial set in less than 40 min), accelerated-set (initial set within 40 min to 1 h 20 min), normal-set (initial set in more than 2 h), and slow-set (initial set in more than 4 h 30 min) types.

The cements may be distinguished by the strength of the stone and the time needed for its attainment, i. e., with a high initial (early) strength and ordinary (standard) ones.

As regards their resistance to aggressive media, the cements are known to be of sulphate-resistant and standard types.

In the USA, there are put out seven classes of plugging cement that are employed for cementing wells of varying depth with high and low bottom-hole temperatures. Some of these cements have to meet demands for a great initial strength, high resistance to the action of sulphates, etc. The principal component in all these classes is portland cement and, therefore, to apply them at high temperatures and pressures, setting retarders must be introduced. Besides basic brands of oil-well cements marketed by the USA industry, pertinent manufacturers produce, in cooperation with research bodies, small amounts of both modified (containing various additions and aggregates) and clinkerless cements that have to conform to specific field conditions.

5.2. Plugging Portland Cement

This is a variety of silicate cement, a product consisting of a mixture made up of ground materials of a specified mineralogical composition. The major part of portland cement is clinker, which is obtained through calcination of a special mixture of limestone and clay (marl) to a point (at a temperature of about 1450 °C) at which all of its constituents are caked. When being calcined, lime (chalk, limestone) yields calcium oxide, while clay is a source of silica (SiO_2), alumina (Al_2O_3), and iron oxide (Fe_2O_3).

To control the structurization of cement slurry and to raise the initial strength of cement stone, 3 to 6 % of gypsum are added to the clinker at the time of its grinding. Also 10 to 15 % of mineral additions, such as slag, tripoli, opoka (gaize), limestone, sand, etc., are introduced into the mix. These serve to improve certain properties of the slurry and stone and help cut down the consumption of costly clinker.

5.3. Clinker Composition

Plugging portland cement is made up of oxides, the principal ones being as follows (in %):

calcium oxide (CaO)	60-66
silica (SiO_2)	18-25
alumina (Al_2O_3)	4-8
iron oxide (Fe_2O_3)	0.5-5

The properties of the plugging mixture and the cement stone vary essentially depending upon the percentages of the aforementioned oxides.

The active properties of plugging portland cement are determined largely by calcium oxide (CaO), chemically bound with silica, alumina and iron oxide.

Silica contributes to the formation of silicates of calcium and aluminium and imparts hydraulic properties to the cement, i. e. the ability to solidify (harden) and to be workable for a long time in an aqueous medium. An increased SiO_2 content brings about some delay in the setting of plugging mixtures under ordinary conditions and raises the resistance of the cement stone to the action of sulphates.

Alumina helps speed up the setting of the cement slurry and reduces the rigidity of the stone.

A higher proportion of iron oxide in the cement leads to slowing down of the processes involved in setting of the plugging mixtures and decreases the early strength of the cement stone.

Among admixtures in the portland cement figure also some other oxides (in %):

MgO	0.1-5.5	TiO_2	0.2-0.5
$K_2O + Na_2O$	0.5-1.3	P_2O_5	0.1-0.3
SO_3	0.3-1.0	MnO	0.5-3.5

When in surplus (over 4.5 %), magnesium oxide (MgO) produces an increase in the volume and disintegration of the cement stone undergoing solidification, this being due to the fact that MgO, being in the clinker in a chemically uncombined state, undergoes a slow hydration (interacts with water), while the mixture has already hardened.

With its content amounting to 4-5 % of the total and a corresponding reduction of the silica (SiO_2) level, titanium dioxide (TiO_2) enhances the strength of the stone stemming from this cement.

Manganese protoxide in an amount of up to 4 % and phosphoric anhydride (P_2O_5) of up to 1-2 % do not affect adversely the properties of the mixture and stone, but under normal conditions P_2O_5 tends to retard the setting.

The sodium and potassium lye content in quantities of more than 0.3-0.4 % is undesirable, for this results in that the setting processes become subject to sharp fluctuations.

Free, i. e. chemically uncombined, calcium oxide is a deleterious component. It appears in the clinker as a result of incomplete clinkering because of an inappropriate compounding of a raw mix, its insufficient homogeneity and incomplete calcination. Free CaO calcinated at a clinkering temperature is hydrated at a much slower rate than do the basic compounds of the clinker. The process of its hydration is attended by an increased volume of its solid phase. When it undergoes hydration in an already solidified cement stone the latter becomes subject to internal stresses leading to non-uniform changes of its volume. To preclude the occurrence of such adverse after-effects the content of free calcium oxide should not be allowed to exceed 1.0 %.

Among harmful constituents of the clinker is also free magnesium oxide (MgO) which also undergoes hydration extremely slowly following calcination at the caking temperature and can cause non-uniform variations in the volume of the cement stone.

When subjected to calcination up to the caking temperature (of about 1450 °C), all of the oxides listed above, while interacting, form artificial minerals, called clinker materials.

According to S. D. Okorokov's classification, silicate cements, and portland cements among them, represent in the mineralogical aspect a mixture of mineral-silicates and fused minerals in a proportion of approximately 3 : 1.

The group of the mineral-silicates includes tricalcium $3CaO \cdot SiO_2$ and dicalcium $2CaO \cdot SiO_2$ silicates.

Among the fused minerals figure the following compounds:

tricalcium aluminate — $3CaO \cdot Al_2O_3$
pentacalcium trialuminate — $5CaO \cdot 3Al_2O_3$
monocalcium aluminate — $CaO \cdot Al_2O_3$
tetracalcium alumoferrite — $4CaO \cdot Al_2O_3 \cdot Fe_2O_3$
monocalcium ferrite — $CaO \cdot Fe_2O_3$

dicalcium ferrite — $2CaO \cdot Fe_2O_3$
Toporov's mineral — $46CaO \cdot 16Al_2O_3 \cdot 7Fe_2O_3$

Besides minerals, silicate cement contains a vitreous mass which is an eutectic melt from which, because of a rapid cooling of the cement clinker, the minerals could not have enough time to separate. The amount of this mass comprises 5-12 per cent. The glass is composed chiefly of non-crystallized ferrites, aluminates, dicalcium silicate, alkaline compounds and of magnesium oxide, partially contained in the clinker.

Among key minerals figure alite and belite. The former consists mainly of tricalcium silicate ($3CaO \cdot SiO_2$). Belite is one of the dicalcium silicate modifications ($\beta = 2CaO \cdot SiO_2$). The intermediate agent is represented largely by celite (calcium alumoferrite). It fills the interspaces between the alite and belite crystals.

The properties of plugging portland cements are determined largely by the presence of the most important minerals, among which are:

tricalcium silicate — $3CaO \cdot SiO_2 (C_3S)$
dicalcium silicate — $2CaO \cdot SiO_2 (C_2S)$
tricalcium aluminate — $3CaO \cdot Al_2O_3 (Ca_3A)$
tetracalcium alumoferrite — $4CaO \cdot Al_2O_3 \cdot Fe_2O_3$

5.4. Quantitative Characteristics of Clinker

The most precise method of determining the composition of clinker is the chemical anlysis. By using it one can determine the content of major oxides, the knowledge of which will make it easy to ascertain (by way of pertinent calculations) the levels of principal minerals and to get an idea about some properties of the portland cement under study. Such an assessment of the portland cement may be made on the basis of its estimated mineralogical composition and by using the saturation coefficient and the modules.

The composition and properties of portland cement depend upon the type of the raw material and on chemical reactions that occur during calcination. With the temperature brought to the maximum level, there take place significant changes in the raw material undergoing calcination.

A properly produced portland cement should contain no more than 0.5 per cent of free lime.

5.5. Saturation Coefficient and Moduli of Portland Cement

As a result of the clinkering processes calcium oxide combines with silica, alumina and iron oxide. This is followed by the formation of corresponding salts, such as silicates, aluminates and calcium ferrites. Since there develop free calcium oxide and free magnesium oxide in the clinker, its constituents in calculating the raw mix should be chosen in such a way as to make it possible for the acid oxides to become bound with adulterants (impurities), i. e. once CaO has been combined completely some of the acid oxides should remain bound, forming less basic compounds. In the course of clinkering the first to be saturated completely with lime are iron and silicon oxides, silica being the only one that remains saturated incompletely.

To assess the degree to which the acid part of the clinker is saturated with lime use is made of saturation coefficient (SC). This represents the ratio between the amount of lime actually bound in the clinker and its theoretically possible quantity for a given content of acid oxides:

$$SC = \frac{CaO - CaO_{free} - 1.65Al_2O_3 - 0.35Fe_2O_3 - 0.7SO_3}{2.8SiO_2 - SiO_{2\,free}} \qquad (34)$$

where CaO_{free} is the calcium oxide that remained unbound (free); $SiO_{2\,free}$ is the unbound (free) silica.

Plugging portland cements have a high saturation coefficient (0.85 to 0.95).

Many years of practical experience helped establish also the other parameters (moduli) that characterize the appropriate chemical composition of cement and the proportions of its major oxides (in %).

The silica modulus is

$$n = SiO_2/(Al_2O_3 + Fe_2O_3) \qquad (34')$$

The alumina modulus is

$$p = Al_2O_3/Fe_2O_3 \qquad (34'')$$

The hydraulic index (rarely used) is

$$m = \frac{CaO}{SiO_2 + Al_2O_3 + Fe_2O_3} \qquad (34''')$$

The following are the limits of changes in the above proportions:

$$n = 1.7 \text{ to } 3.5; \quad p = 1.0 \text{ to } 3.0; \quad m = 1.9 \text{ to } 2.4$$

5.6. Estimated and Actual Mineralogical Composition of Portland Cement Clinker

The mineralogical composition of portland cement clinker may be calculated (under certain assumptions) from the content of oxides by using a procedure proposed by V. A. Kind.

In concise writing of oxides contained in the clinker, as adopted in the cement chemistry, the designations differ from the generally accepted ones, namely: CaO is denoted by the letter C; SiO_2 — by S; Al_2O_3 — by A; Fe_2O_3 — by F and H_2O — by H.

When computing the mineralogical composition it is assumed that the clinker contains only pure minerals C_3S, C_2S, C_3A, C_4AF; that the aluminate phase is represented by C_3A; sulphates — by calcium sulphate. In the calculation are involved only the principal oxides — CaO, SiO_2, Al_2O_3, Fe_2O_3 and SO_3, and the process is attended by separation of free lime (CaO) and unbound silica (SiO_2). It is assumed that the vitreous phase is absent altogether.

The mineralogical composition of the portland cement clinker estimated with certain assumptions is a conventional one.

It is assumed that the first compound that forms during calcination of the raw material is C_4AF, which includes the whole of the Fe_2O_3. The stoichiometric relations may be written down as follows:
molecular quantities

$$4CaO + Al_2O_3 + Fe_2O_3 = 4CaO \cdot Al_2O_3 \cdot Fe_2O_3$$

quantities by mass

$$(4 \times 56) + 102 + 159 = 485$$

Hence, one mass percent of Fe_2O_3 combines

$$\frac{4 \times 57}{159} = 1.4\ \%\ \text{CaO} \quad \text{and} \quad \frac{102}{159} = 0.64\ \%\ Al_2O_3$$

This gives rise to the formation of $485/159 = 3.04\ \%$ of C_4AF of the clinker's mass.

Then, the amount of tetracalcium alumoferrite may be determined from the formula

$$C_4AF = 3.04\ \%\ F$$

where F is the percentage of Fe_2O_3 in the clinker.

As regards the remaining alumina, we may write the following:
molecular quantities

$$3CaO + Al_2O_3 = 3CaO \cdot Al_2O_3$$

quantities by mass

$$(3 \times 56) + 102 = 270$$

Consequently, each mass percent of the remaining Al_2O_3 combines $3 \times 56/102 = 1.65\ \%$ of CaO. This is attended by the formation of $270/102 = 2.65\ \%$ of C_3A. Then, to determine the proportion of tricalcium aluminate we shall write the formula

$$C_3A = 2.65\,(A - 0.64F)$$

Some of calcium oxide is bound by sulphate:

$$CaO + SO_3 = CaSO_4\ (56 + 80 = 136)$$

i. e., each mass percent of SO_3 combines $56/80 = 0.7\ \%$ of CaO. This is attended by the formation of $1.7\ \%$ of $CaSO_4$.

Then we may write

$$CaSO_4 = 1.7\ \%\ SO_3$$

Further, the amount of the remaining lime can be estimated.

To form calcium silicates (C_3S and C_2S)

$$CaO - 1.4F - 1.65\,(A - 0.64F) - 0.7SO_3 = CaO - 1.65Al_2O_3 - 0.35Fe_2O_3 - 0.7SO_3$$

For C_2S, which forms prior to the development of C_3S, the amount of CaO will be determined in the following way. At first the whole of silica reacts by forming dicalcium silicate

$$2CaO + SiO_2 = 2CaO \cdot SiO_2 \; (2 \times 56 + 60 = 172)$$

i. e. a single mass percent of silica yields $172/60 = 2.86$ % of C_2S and is bound with 1.87 % of CaO.

To form the tricalcium silicate C_3S there remains

$$CaO - 1.65Al_2O_3 - 0.35Fe_2O_3 - 0.7SO_3 - 1.87SiO_2$$

Let us write the reaction

$$2CaO \cdot SiO_2 + CaO = 3CaO \cdot SiO_2 \; (172 + 56 = 228)$$

i. e., one mass percent of the remaining CaO yields $228/56 = 4.07$ % of C_3S.

Now we can determine the quantity of the developing tricalcium silicate $3CaO \cdot SiO_2\,(C_3S)$

$$C_3S = 4.07\,(CaO - 1.65Al_2O_3 - 0.35Fe_2O_3 - 1.87SiO_2 - 0.7SO_3)$$

On one mass percent falls $60 : 228 = 0.26$ % of SiO_2. The remaining amount of SiO_2 is spent in forming C_2S

$$C_2S = 2.86[SiO_2 - 0.26(4.07CaO - 6.72Al_2O_3 - 1.42Fe_2O_3 - 2.84SO_3 - 7.6SiO_2)] =$$
$$= 8.6SiO_2 + 5.07Al_2O_3 + 1.07Fe_2O_3 + 2.15SO_3 - 3.07CaO$$

The earlier adopted admissions make the estimated mineralogical composition of the clinker to differ substantially from the actual one. The actual amount of alite in the commercially produced clinker is higher and that of belite is lower than envisaged by the estimate.

A higher content of the mineral-silicates and a greater strength of portland cement stone come from an increase of the silica modulus. Should there be a concurrent fall of the saturation coefficient, the strength of cement stone will decrease. Greater setting rates are usually due to an increased alumina modulus.

5.7. Brief Information on the Technology of Portland Cement Production

In the production of portland cement the best raw material is considered to be marl. It is most desirable that the ratio between the lime and clay components of the marl employed should closely approach that commonly adopted in the manufacture of portland cements (roughly 78 : 22). Should marls be lacking altogether, or the composition of the available ones differ from that specified, calcium carbonates are made use of as a lime constituent. The proportion of magnesium carbonate in calcium carbonates must not exceed 2.0-2.5 per cent.

As an argillaceous component it is kaolinite constituents with the habitual levels of 40-60 % of SiO_2, 10-20 % of Al_2O_3, 4-8 % of Fe_2O_3 and 2-12 % of CaO that are put to use more often than not. The alkali metal oxides, often present in clays, are noxious impurities and their content should be kept down below 5 per cent. In place of the argillaceous component (loam, loess, shales) use is often made of granulated blast furnace slags and nepheline sludge, to which must be added adequate components to produce portland cement.

In making up an artificial raw mix or treating natural marls even with their composition being close to the required one, adjusting additions should be introduced. These include materials that commonly contain some acid oxides (SiO_2, Al_2O_3, Fe_2O_3). When granulated blast furnace slags or nepheline sludge are utilized, some amount of CaO (15-20 %) is added. By varying the proportions of the argillaceous and calcareous constituents it is possible to change the content of the oxides group, which does not always lead to the desired result. For this reason, adjusting additions in the form of individual acid oxides are introduced. Silica is commonly introduced together with opoka and quartz sand, while alumina with bauxite, and iron oxides with iron ore, pyrite cinders, furnace shaft dust.

The portland cement production flow-sheet. Two processes — dry and wet — are employed in the manufacture of portland cement. With the former the raw material components, after their preliminary drying, are proportioned and ground in mills to powder with the grain size of up to 100 μ. The mix is then adjusted and homogenized pneumatically and afterwards calcined in special kilns. There exist several variants of preheating the mix up to a temperature of 900-1000 °C. Thereafter it is brought into an inside-lined revolving furnace (with rotational velocity of 1 rpm).

On cooling in the revolving furnace the clinker is ground. In the course of milling gypsum, slag, sand or other additives are intermingled with the clinker and it is this final powder that is known by the name of cement.

Widely popular has become the wet method of cement manufacture. In this case the grinding and adjustment of the raw mix is attended by an addition of water. The mix is readily homogenized through intermingling (agitation), pumping-over or by other methods. Inasmuch as the mix appears as a gelly-like paste, its transportation, adjustment and stirring are greatly facilitated. The mix is calcined (roasted) in long (up to 200 m) revolving furnaces with their diameter reaching 5 m. Upon cooling the mix is ground in ball mills. When milling goes on, additives raising the physico-mechanical properties of the portland cement are introduced.

The Soviet cement plants put out several varieties (brands) of portland cement.

Plugging portland cements for "cold" and "hot" wells belong to the category of active quick-setting cements.

5.8. Properties of Dry Cement Flour

Unlike other construction materials the properties of portland cements are verified on hardening of the cement mixture, although they are envisaged during production of the cement.

Of paramount importance are the following properties of portland cement.

The density of portland cement. Of all the binding agents, portland cement has the greatest density, averaging 3.12 to 3.15 g/cm^3. The density of plugging portland cements is indicative of the properties of plugging mixtures prepared from them, because the density of the mixtures depends on that of the cements. The density of plugging cements is also indicative of the presence in them of lightening or weighting additives.

In use is also what is known as unit bulk mass (i. e., the mass of 1 liter of cement flour). It ranges from 900 to 1100 g/l for loose cement flour and from 1400 to 1700 g/l for compacted flour. This quantity is used in planning cement storage facilities and determining the hopper capacity of cement mixers.

The fineness of portland cement. This is a very important property not only of portland cement, but of all special-purpose cements. The fineness of cement flour is determined by screening with reference to the sieve residue and by employing special devices that help establish the specific surface, i. e., the total surface area of particles contained in 1 g of cement. This is given in terms of cm^2/g.

The sieve analysis enables one to quickly assess the granulometric composition (grain-size distribution) of the cement flour. It is common practice to determine the relative content of the fraction measuring over 70-90 μ and the grain fractions of a smaller size. To do this, use is made of the No. 008 sieve with a clear size of 80 μ. A sieve with 4900 apertures over an area of 1 cm^2 is also employed. In this case the mesh size is 88 μ.

While yielding the same sieve residue, different types of cement may greatly differ in the amount of the finest fractions, this being determined by the grindability of the clinker and additives, the nature and the quantity of the aggregate, grinding technology, etc. The grain-size distribution in ordinary brands of portland cement is roughly as follows: 30 to 40 % particles less than 10 μ in grain size, 10 to 20 % particles 10 to 20 μ in grain size, 10 to 20 % particles 20 to 30 μ in grain size, 10 to 20 % particles 30 to 50 μ in grain size, and 10 to 25 % particles more than 50 μ in grain size. Coarse fractions contain an elevated amount of belite and ferrite constituents (since these represent the more difficultly crushed components), while the fine-grained fractions (fines) carry an increased proportion of alite.

It is the finest fractions that influence most the characteristics of cement. Their summary surface is by far superior to that of the particles in the coarser fractions, though their share in the total mass is not great. The smallest size of the portland cement grains measured by means of an electron microscope is approximately 0.5 μ.

The specific surface of cement and of other powderlike materials employed in oil-well cementing is determined with the ПСХ-2 Model apparatus. Its operation is based on the measurement of the surface of powderlike materials by the method of airtightness. There are also known other apparatus operating on the same principle (methods proposed by V. V. Tovarov, R. Blaine, Lee and others).

Plugging portland cements have a specific surface equalling roughly 2500-4000 cm^2/g. In the course of grinding, active mineral agents are added to the clinker.

5.9. Active Mineral Additives to Binders

According to the standard definition, by the name of active mineral additives are meant substances (natural or artificial) which, when mixed in a finely-ground form with air-slaked lime and with water, produce a paste able, upon hardening in the air, to continue hardening under water as well. Additives are used in producing diverse types of cement.

Active mineral additives are classed in the following two groups: Group I — natural additives and Group II — artificial additives.

Group I Additives

A. Additives of sedimentary origin.

(a) Diatomites — rocks consisting chiefly of agglomerations of microscopic frustules which contain largely amorphous silica.

(b) Tripoli — rocks made up of microscopic, mainly rounded, grains with amorphous silica as their principal component.

(c) Opoka — compacted diatomite and tripoli.

(d) Glaize (baked clay) — rocks formed as a result of natural roasting of clay during underground fires in coal seams.

B. Additives of igneous origin.

(a) Ash — rocks containing alumosilicates and found in nature in the shape of loose, partly consolidated deposits.

(b) Tuffs — consolidated and cemented (coherent) igneous rocks.

(c) Pumice — lithoidal rocks characterized by their porous, spongy structure (swallen volcanic glass).

(d) Trass — metamorphized varieties of volcanic tuffs.

Group II Additives

(a) Siliceous waste — rejects rich in silicic acid, obtained during extraction of alumina and clay.

(b) Calcined clays — a product of artificial roasting of argillaceous rocks and of self-igniting mine waste-pile rocks (clay and coaly shales).

(c) Ash, cinder and boiler slags — a solid by-product that forms on burning at a definite temperature of some types of fuel, whose mineral component abounds in acidic oxides.

(d) Granulated blast-furnace slags (acid and basic ones) — silicate melts that emerge in smelting of pig-iron and assume a fine-grained structure through their rapid cooling.

5.10. Heat Liberation During Hardening of Plugging Mixtures

Hydration of portland cement is an exothermal process accompanied by liberation of heat.

Of paramount importance in the heat evolution of cements is their chemico-mineralogical composition, which, in the end, determines the rate of their hydration. To a lesser degree the thermochemical properties are affected by other factors, such as the fineness of grinding, the period and conditions of storage, the grinding method, etc. As regards the magnitude and the nature of heat evolution development in time, clinker materials are arrayed in the following order:

$$C_3A > C_3S > C_4AF > C_2S$$

In hardening, tricalcium aluminate and tricalcium silicate evolve heat initially at the highest rate, while dicalcium silicate displays first an insignificant heat liberation which substantially intensifies later on.

In hardening of plugging portland cement mixtures, the greatest amount of heat is evolved during the first three-five days. With rising ambient temperature the period of the most intensive emission of heat is seen to decrease.

An extremely high rate of heat liberation is common to the alumina cement; after three-four days of hardening at ordinary temperature the amount of heat emitted by it reaches the maximum level.

The heat liberation effect can be brought down through introduction into cements of large quantities of aggregates, but this is apt to materially change other physico-mechanical characteristics of the mixture and stone.

Proceeding from general propositions it may be noted that some additives belonging to the group of plasticizers (spent sulfite-alcohol liquor, for instance) envelop the grain surface, thus forming films of a definite thickness, and at a certain temperature they don't let the water pass through for some time and thereby retard the processes of hydrolysis and hydration of the cement grains. In this particular case the initial heat liberation will be kept down. With the passage of time, because of the breakdown of the surfactants' films, the hardening and heat evolution processes will be proceeding virtually in a normal manner.

Application of reagents-accelerators ($CaCl_2$, for example) leads to a noticeable acceleration and enhancement of the thermal effect in the hardening cement slurry stone.

With increasing temperature the heat liberation rate rises too. In portland cement mixtures undergoing hardening, an increase of the pressure is followed by a greater evolution of heat. A change of the water-cement ratio from 0.3 up to 0.6 at a pressure of 5 to 30 MPa tends to markedly speed up the process of heat liberation.

Practical Studies

1. By using formulas (34), calculate *SC*, *n*, *p*, and *m* on the basis of the known chemical composition of portland cement.

2. By making use of the procedures described in Sec. 5.6, estimate on the grounds of the known chemical composition (content of oxides) the mineralogical composition of clinker.

Laboratory Work No. 2

I. Determination of physical properties of cement flour.

1. Determine density of cement flour.

The density of cement flour is determined by using the Le Chatelier apparatus (Fig. 46).

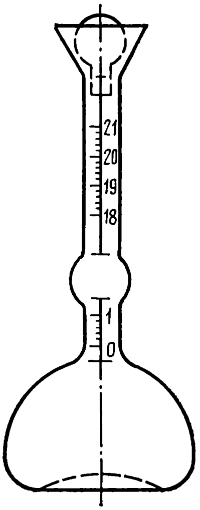

Fig. 46. Le Chatelier apparatus for determining the density of cement flour

Put the apparatus into a glass vessel so as to have its calibrated portion submerged in water. To prevent its coming to the surface, fasten the apparatus in a holder. When reading the level of the fluid in the apparatus the temperature of water in the vessel should accord with that at which the apparatus was calibrated.

Fill the apparatus to the lower zero line with anhydrous kerosene, the bottom meniscus serving as a reference. Thereafter wipe dry thoroughly the part of the apparatus free of kerosene (above the zero line) with a filter paper wad.

Weight out 65 g of cement to an accuracy of 0.01 g, preliminarily dried for 2 hours at 105-110 °C in an exsiccator and then cooled therein. Through a funnel pour into the apparatus with a spoon small uniform lots of the cement till the level of the fluid in the apparatus reaches one of the readings within the upper calibrated part of the apparatus. To remove air bubbles, withdraw the apparatus with its contents from the vessel with water and keep it in a tilted position on a smooth rubber rug for 10 min. Thereupon place the apparatus anew for not less than 10 min into water whose temperature equals that at which the apparatus was calibrated and read off the level of the fluid in the apparatus.

The density (ρ_c) of cement flour (in g/cm^3) is calculated from the formula

$$\rho_c = g/V$$

where g is the weighed amount of cement, g; V is the volume of fluid displaced by cement flour, cm^3.

The density of the cement flour tested is estimated to an accuracy of up to 0.01 g/cm^3 as the arithmetical mean of the results of two measurements whose divergence should not exceed 0.02 g/cm^3.

II. Determination of the specific surface of cement.

The specific surface of the cement flour may be determined by the method of air-tightness.

The general arrangement of the apparatus for determining the specific surface of the cement is shown in Fig. 47. The apparatus consists of five parts, *viz.*, case *1*, pressure gauge-aspirator *2*, valve *3*, pressure (rarefaction) regulator *4*, and rarefaction means (bulb or water-jet pump) *5*.

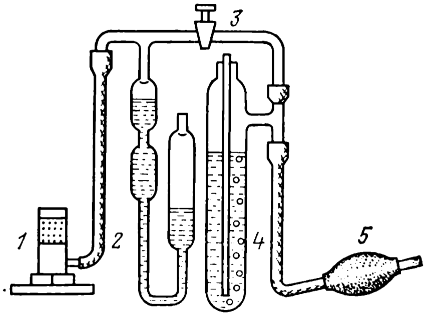

Fig. 47. Apparatus for determining the specific surface of cement flour

Before testing, screen the cement through a No. 09 sieve and then desiccate the weighed amount of 25 g for 2 hours at 105-110 °C in a drying cabinet.

Check on the hermetic sealing of the case and connections of the apparatus. To do this, tightly close the top of the case with a rubber plug, create rarefaction in the pressure gauge-aspirator and shut off the valve. With complete tightness of the case and connections the level of the fluid in the pressure gauge-aspirator should not go down. Otherwise the site of the air in-seepage is to be found and then the complete tightness of the apparatus secured.

The mass Q in grams (accurate to within 0.01 g) of the sample batch of cement is found from the formula

$$Q = \rho_c V (1 - m)$$

where ρ_c is the density of the cement to be tested, g/cm^3; V is the volume of the cement layer in the case, cm^3; m is the voids ratio, fractions of unity (recommended to be taken at 0.48 ± 0.01).

In case of portland cements ground without any additives (except for gypsum), the density may be taken to equal 3.15.

For very finely ground cements, as well as for the ones carrying additives (silica, carbonate and other brands) the weight of the specimen batch is to be selected by the trial and error method, so as to obtain a compact layer after the material has been pressed in the case with a plunger. Then the voids ratio is calculated according to the formula

$$m = (V\rho_c - Q)/V\rho_c$$

To prepare the cement layer for testing put a perforated disk into the case. On top of the disk place a ring of a low- or medium-density filter paper carrying a weighed amount of the cement specimen. Lightly tap on the case walls, put on top of the cement layer a second ring of filter paper and press the specimen with a plunger pushed in by hand for as long as the thrust ring of the plunger contacts the top edge of the case.

By using a rubber tube, connect the case with the prepared cement layer to the pressure gauge-aspirator.

With the water-jet pump of some other means of rarefaction set going, turn off the valve as soon as the fluid in the closed elbow of the pressure gauge-aspirator attains the height between the two lines marked on the tube. Variations in the level of the fluid in the closed elbow of the pressure gauge-aspirator causes seepage of the air through the layer of the material in the case. When the level of the fluid reaches the mark above the upper widening, start a timer and, when the level of the fluid reaches a point between the two widenings, stop it.

Should the level of the fluid be falling too rapidly, this making it impossible to precisely note the time of the fluid meniscus passing the first mark (above the upper widening), use the lower widening of the pressure gauge-aspirator for making the measurements. In this case set going the timer when the fluid level reaches the line between the widenings and stop it when the fluid level declines to a mark lying below the second widening.

Measure twice the duration of the air seepage for one and the same weighed batch and in further calculations take an arithmetic mean of two measurements.

In measurements requiring high precision, make two-three repeated estimations by taking separate weighed amounts of the cement.

Specific surface S of the cement, in cm^2/g, is calculated by using the formula

$$S = \frac{k}{\rho_c} \sqrt{m^3/(1-m)^2}\ \sqrt{1/h}\ \sqrt{T}$$

where k is the constant of the apparatus indicated in its certificate separately for dealing with the upper and lower widenings of the pressure gauge-aspirator; ρ_c is the density of the cement, g/cm^3; m is the voids ratio of the cement in the case, fractions of unity; T is the time passed during the drop of the fluid level from the mark at the top of the widening down to the point at the bottom of the widening, s; h is the air viscosity at the test temperature, Pas. Numerical values of $\sqrt{m^3/(1-m)^2}$, depending upon m, are listed in Table 17 and numerical values of $\sqrt{1/h}$ at various temperatures, in Table 18.

Table 17

Numerical Values of $\sqrt{m^3/(1-m)^2}$ as a Function of m

m	$\sqrt{m^3/(1-m)^2}$	m	$\sqrt{m^3/(1-m)^2}$	m	$\sqrt{m^3/(1-m)^2}$	m	$\sqrt{m^3/(1-m)^2}$
0.450	0.549	0.476	0.627	0.501	0.711	0.526	0.805
0.451	0.552	0.477	0.630	0.502	0.714	0.527	0.809
0.452	0.554	0.478	0.633	0.503	0.718	0.528	0.813
0.453	0.557	0.479	0.636	0.504	0.721	0.529	0.817
0.454	0.560	0.480	0.639	0.505	0.725	0.530	0.821
0.455	0.563	0.481	0.643	0.506	0.729	0.531	0.825
0.456	0.566	0.482	0.646	0.507	0.733	0.532	0.829
0.457	0.569	0.483	0.649	0.508	0.736	0.533	0.833
0.458	0.572	0.484	0.652	0.509	0.739	0.534	0.837
0.459	0.575	0.485	0.656	0.510	0.743	0.535	0.842
0.460	0.578	0.486	0.659	0.511	0.747	0.536	0.846
0.461	0.581	0.487	0.662	0.512	0.751	0.537	0.850
0.462	0.584	0.488	0.666	0.513	0.755	0.538	0.854
0.463	0.587	0.489	0.669	0.514	0.758	0.539	0.858
0.464	0.590	0.490	0.672	0.515	0.762	0.540	0.863
0.465	0.593	0.491	0.676	0.516	0.766	0.541	0.867
0.466	0.596	0.492	0.679	0.517	0.770	0.542	0.871
0.467	0.599	0.493	0.683	0.518	0.774	0.543	0.875
0.468	0.602	0.494	0.686	0.519	0.777	0.544	0.880
0.469	0.605	0.495	0.690	0.520	0.781	0.545	0.884
0.470	0.608	0.496	0.693	0.521	0.785	0.546	0.889
0.471	0.611	0.497	0.697	0.522	0.789	0.547	0.893
0.472	0.614	0.498	0.700	0.523	0.793	0.548	0.898
0.473	0.617	0.499	0.704	0.524	0.797	0.549	0.902
0.474	0.620	0.500	0.707	0.525	0.801	0.550	0.906
0.475	0.624						

When determining the specific surface of one and the same material of constant density (e. g., during process of control over fineness of cement leaving a mill, for instance) it is recommended to measure the surface at a constant value of the voids ratio. The computation is then made according to the formula

$$S = A\sqrt{T}$$

where

$$A = k/\rho_c \sqrt{m^3/(1-m)^2}\sqrt{1/h}$$

Table 18

Numerical Values of $\sqrt{1/h}$ at Various Temperatures

Temperature, °C	Density of mercury, g/cm³	Viscosity of air, 10^{-8} Pa s	$\sqrt{h}$	$\sqrt{1/h}$
8	13.58	1749	0.01322	75.64
10	13.57	1759	0.01326	75.41
12	13.57	1768	0.01329	75.21
14	13.56	1778	0.01333	75.00
16	13.56	1788	0.01337	74.79
18	13.55	1798	0.01342	74.58
20	13.55	1808	0.01344	74.37
22	13.54	1818	0.01348	74.16
24	13.54	1828	0.01352	73.96
26	13.53	1837	0.01355	73.78
28	13.53	1847	0.01359	73.58
30	13.52	1857	0.01362	73.38
32	13.51	1867	0.01366	73.19
34	13.51	1876	0.01369	73.01

An Example of Calculation

Basic data. The volume of the cement layer in the case — 7.5 cm³; the density of cement — 3.1 g/cm³; weighed amount (sample batch) of cement — 12 g; the constant of the apparatus $k = 25$ (of the upper widening); the duration of the test, $T = 63$ s; test temperature — 20 °C.

Calculation

The voids ratio

$$m = \frac{7.5 \times 3.1 - 12.0}{7.5 \times 3.1} = 0.484$$

$$\sqrt{m^3/(1-m)^2} \text{ (from Table 17)} = \sqrt{\frac{0.484^3}{(1-0.484)^2}} = 0.652$$

$$\sqrt{1/h} \text{ (from Table 18)} = 74.37$$

$$S = \frac{25.0}{3.10}\, 0.652 \times 74.37 \sqrt{63} = 3100 \text{ cm}^2/\text{g}$$

With $\rho_c = 3.1$, $m = 0.48$ and the air temperature in the range 13 to 19 °C, the value of A will be

$$A = \frac{25}{3.1}\, 0.652 \times 74.37 = 390$$

$$S = 390\sqrt{T}$$

Note. The results of calculations should be rounded off to form integral tens of cm²/g.

III. Determination of cement flour fineness.

The following equipment is used in determining the fineness of cement: (a) a sieve with No. 008 screen (the mesh size in clear is 0.08 × 0.08 mm). The screen should be drawn tight and firmly pressed inside a cylindrical casing 10-15 cm in diameter; (b) an appliance for mechanical screening of cement. In the absence of such a device in the laboratory the screening may be done by hand.

Weight out 50 g of cement, preliminarily desiccated in a drying cabinet for 1 hour at 105-110 °C, and then pour it out onto a sieve. Having closed the latter with a cover, mount the sieve on the device for mechanical screening. Approximately 5-7 minutes from the start of screening arrest the sieve, carefully remove the bottom and pour out therefrom the cement that has passed through the sieve. To reduce the clogging of the sieve during subsequent screening clean the underside of the sieve with a soft brush, insert the bottom part and resume screening.

The control screening is to be done onto a sheet of paper with the removed bottom part. The screening is considered completed when with manual screening no more than 0.05 g of cement passes through the sieve in 1 minute. The sieve should be absolutely dry and is to be cleaned after testing.

The cement fineness is determined to an accuracy of up to 0.1 % in terms of a residue on the sieve with a screen mesh No. 008 in per cent to the initial weight of the sample undergoing screening.

IV. Determination of the unit bulk mass of dry cement.

Pour out from a height of 10 cm an average sample lot of cement dried to a constant weight into a preliminarily weighed measuring vessel of 1 litre capacity (the vessel commonly used for this purpose has a diameter and height of 234 mm) for as long as a cone is formed over and above the vessel. Cut the surplus cement with a ruler flush with the brim of the vessel (but without compacting the mass). Thereupon weigh the vessel together with the cement flour. The unit bulk mass of dry cement ρ_b is calculated in kg/m^3 to an accuracy of up to 10 kg/m^3 from the formula

$$\rho_b = (g_2 - g_1)/V \qquad (35)$$

where g_1 is the mass of the measuring vessel, kg; g_2 is the mass of the measuring vessel with cement, kg; V is the capacity of the measuring vessel, m^3.

The unit bulk mass of dry cement is taken as the arithmetic mean of three measurements. In determining the unit bulk mass of cement in its natural state, the same procedure, but without any preliminary drying of cement, is adopted.

V. Chemical analysis of cement.

The following constituents are determined in cements: SiO_2, Fe_2O_3, Al_2O_3, CaO, MgO, SO_3, the sum of $K_2O + Na_2O$ and losses during calcination (l. d. c.). The sum total of the alkali and losses incurred during calcination are found from individual weighed lots and the other components — from a single weighed lot.

Fe_2O_3, Al_2O_3, CaO and MgO are determined complexometrically, while SiO_2 and SO_3 by the mass method.

Determination of losses during calcination (*l. d. c*). Place a lot of cement weighing 1.0 g into a porcelain crucible, preliminarily calcined to a constant mass, calcine in a muffle at 900-1000 °C for 1 hour, cool in an exsiccator and weigh. Repeat calcination until constant mass. Repeated calcinations should last 20 minutes each time.

The l. d. c. (in %) are found from the formula

$$\text{l. d. c.} = (v \times 100)/a$$

where v is the difference in the crucible mass together with the sample lot before and after calcination, g; a is the weighed sample lot of cement, g.

Determination of SiO_2. Place a sample lot of cement weighing 0.5 g, finely ground and dryed at 105 °C, into a platinum crucible, intermix with a six-fold amount (3.0 g) of flux *, close with a cover and melt in a muffle at 900-1000 °C.

Cool the crucible with the melt in a bowl with distilled water. Mount an evaporating dish containing 100 ml of hydrochloric acid diluted 1 : 3 on a water bath **. Upon its cooling transfer the crucible into the evaporating dish and quickly cover the latter with a glass plate so as to prevent splashing. Once the leaching is completed, flush the glass, the crucible and the cover with hot distilled water from a washing bottle. Add one-two drops of nitric acid to the solution. Boil down dry the contents of the dish, treat with hydrochloric acid and then boil down again. Repeat the hydrochloric acid treatment 2-3 times. Upon dehydration take the dish off the bath, allow the contents of the dish to cool down, thoroughly triturate the lumps with a glass rod, pour into the dish 10 ml of HCl with a density of 1.17 g/cm^3 and 30 ml of hot distilled water. Transfer the contents of the dish onto a filter, wash the residue 3-4 times with a hot flushing solution (HCl: distilled water in the ratio 1 : 95), first through decantation and then on the filter.

Complete washing of the residue *** with hot distilled water till disappearance of the chlorine ions ****. Put the residue with the filter into a porcelain crucible, incinerate on an electric stove, calcine in a muffle at 900 °C for 1 hour, put into an exsiccator for cooling and weigh. Calcine to constant mass. Repeated calcinations last 20-25 minutes each time.

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* Two parts of anhydrous sodium carbonate mixed with one part of sublimated borax. To 100 g of the mixture is added 0.5 g of KNO_3. The mixture is to be thoroughly stirred.

** Bunsen beakers made of fire-proof glass are convenient for use as a water-bath.

*** The residue should be washed with small lots of water, the solution allowed to flow down and the washing repeated once more.

**** The test for the chlorine ion is effected as follows: take from a measuring funnel a few drops of the solution into a test-tube, and one drop of HNO_3 and one drop of $AgNO_3$. Should the residue not be washed free from chlorine a milky-white cloud will appear in the solution.

The calculation is made according to the formula

$$SiO_2 = (v \times 100)/a$$

where v is the mass of the calcined residue, g; a is the cement sample lot, g.

Separation of sesquioxides (Fe_2O_3, Al_2O_3) *from calcium and magnesium.* Following separation of silicic acid, boil the filtrate down to a volume of 100 ml, add one-two drops of methyl red and small lots of ammonia till the colouration changes from pink to yellow. Allow the residue to settle on a warm (but not hot) range and pass it through a filter with a red tape. Wash the residue 4-5 times with warm water. Transfer the filtrate into a measuring flask of 500 ml capacity, let it cool down and add distilled water to the mark. Determine CaO, MgO and SiO_2 in the filtrate.

Put the residue on the filter together with the funnel into a 250 ml conical flask (beaker), dissolve in hot hydrochloric acid 1 : 3, thoroughly flush the filter with hot distilled water. The resultant solution serves the purpose of determining iron and aluminium.

Determination of iron oxide. The complexometric determination of iron (III) is done in a muriatic solution with pH from 0.8 to 1.2 in the presence of sulfosalycilic acid serving as an indicator. Sulfosalycilic acid forms with iron ions a violet-red coloured complex which, when titrated with the complexon III, turns into an iron complexonate of a greenish-yellow colour, demonstrable at the equivalence point.

When determining iron in the presence of aluminium one should strictly adhere to the limits of the pH value mentioned above, for with a lower pH value the iron is apt to become titrated incompletely, while with a higher value it is aluminium that begins to undergo titration.

Reagents

1. Hydrochloric acid, diluted 1 : 1.
2. Hydrochloric acid, 1 normal solution.
3. Hydrous ammonia, 25 % solution.
4. Complexon III, 0.05 M solution.

The solution is prepared with fixanal.

The complexon solution titre is fixed with reference to a standard solution of zinc chloride.

To do this, draw off with a pipette 10 ml of a complexon solution, dilute with water to a volume of 100 ml, add 5 ml of a buffer solution with pH of about 5, one-two drops of a 0.5 % aqueous solution of xylenol orange and titrate with a standard solution of zinc chloride till the yellow colouration of the solution turns into reddish one.

The molarity D_c of the complexon solution is found from the formula

$$D_c = D_{Zn}A_{Zn}/10$$

where D_{Zn} is the molarity of the standard zinc solution; A_{Zn} is the quantity of the zinc solution used up in titration, ml; 10 is the amount of the complexon solution taken for titration, ml.

During the complex-formation reaction proceeding under above conditions, one molecule of the complexon III is used up per one ion of a tri- or bivalent metal. Hence, the titre of the complexon solution with reference to iron oxide ($T_{Fe_2O_3}$) is calculated according to the formula

$$T_{Fe_2O_3} = D_c \frac{159.70}{2} \times \frac{1}{1000} = D_c 0.07985 \text{ g/ml}$$

where D_c is the molarity of the complexon III solution, established with reference to a standard zinc solution; 159.70 is the relative molecular mass of iron oxide.

5. Indicator paper "Congo".
6. Sulfosalycilic acid, 25 % solution.

The assay procedure. Heat the muriatic solution obtained through dissolution of the hydroxides residue to a temperature of 70 °C and cautiously neutralize with a 25 % ammonia by watching the indicator paper "Congo" for it to assume brown colouration, or until there emerges a cloud failing to disappear upon stirring, which is then dissolved in one-two drops of hydrochloric acid (1 : 1). To the solution neutralized in this way add 9-10 ml of 1 normal hydrochloric acid (to bring pH up to 1) and distilled water to a volume of 100 ml and heat up to 50-55 °C (in cooler solutions the change of the colouration is drawn out in time, while in the hotter ones there can occur partial titration of aluminium). Further, add three-five drops of a 25 % solution of sulfosalycilic acid and titrate with a 0.05 M complexon III solution till the violet-red colouration of the sulfosilycic iron assumes a greenish-yellow one, characteristic of the iron complexonate. The iron oxide content (Fe_2O_3) in the cement under study is found from the formula (in per cent)

$$Fe_2O_3 = \frac{T_{Fe_2O_3} \times 100}{a} V$$

where V is the quantity of the complexon III solution used up in titration, ml; a is the weighed sample lot of cement, g.

Determination of aluminium oxide (Al_2O_3). The method is based on an indirect complexometric determination of aluminium. The hampering effect of most cations in complex mixtures is eliminated by adding an excess complexon III, which is retitrated with a zinc salt solution. Upon addition to this solution of an excess of sodium fluoride the aluminium is displaced from the complexonate and precipitates on boiling in the form of cryolite. The complexon III released in an amount equivalent to the aluminium content is titrated for the second time with zinc salt solution at pH = = 5.2-5.8, built up by means of an acetate buffer.

It has been ascertained that the content of up to 61.5 per cent of CaO in a sample does not interfere with determination of aluminium, for with pH = 5.2-5.8 calcium does not become bound with the complexon III. In this case a complete liberation of aluminium from the complexonate is greatly influenced by an added amount of sodium fluoride, this amount depending not only on the aluminium content but also upon that of calcium. The latter precipitates in the form of CaF_2 prior to deposition of cryolite. In estimating the level of aluminium use is made of an empiric titre of the zinc salt solution with reference to aluminium oxide.

The empirical titre of the zinc chloride solution is equal to the theoretical one (1 ml of a 0.05 *M* zinc chloride solution corresponds to 0.0025485 g of aluminium oxide) multiplied by the coefficient 1.039. Thus, 1 ml of a 0.05 *M* zinc chloride solution corresponds to 0.002648 g of aluminium oxide.

Reagents

1. A 0.05 *M* zinc chloride solution. Dissolve by heating 3.269 g of metallic zinc "without arsenic" in 28 ml of hydrochloric acid with specific weight of 1.12 and, adding water, adjust the volume of the solution to 1 litre.
2. Complexon III, a 0.05 *M* solution.
3. An acetate buffer solution with pH = 5.2-5.8. Dissolve 250 g of sodium acetate in water, add 20 ml of glacial acetic acid and by pouring in water bring the total volume up to 1 litre.
4. A 0.5 % aqueous solution of xylenol orange.

The assay procedure. On titration of iron neutralize the solution with reference to the "Congo" paper till brown colouration, pour in from a burette 15-20 ml (depending upon the content of Al_2O_3 in the solution) of a 0.05 *M* complexon III solution, 20 ml of a buffer acetate solution, heat up to the bubble point and boil for approximately 3 minutes. Introduce two-three drops of 0.5 % aqueous solution of xylenol orange into the cooled solution and titrate with a 0.05 *M* zinc chloride solution for as long as the colouration of the solution changes from yellow to orange. The quantity of the zinc solution used up in binding the excess of the complexon III is further left out of the calculations. Thereafter, add 40 ml of a saturated sodium fluoride to the solution. Boil it for 3 minutes and then put aside till complete cooling. Add one-two more drops of the xylenol orange to the cooled solution and titrate the liberated complexon with a 0.05 M zinc chloride solution for as long as the yellow colouration turns orange. The spent quantity of the zinc chloride solution corresponds to the amount of aluminium oxide.

The aluminium oxide content (in %) is calculated from the formula

$$Al_2O_3 = \frac{0.002648V}{a} \times 100$$

where 0.002648 is the titre (empirical) of a 0.05 M zinc chloride solution in terms of grams of aluminium oxide.

Determination of calcium oxide. The complexometric determination of calcium oxide is based on its capacity to be titrated with the complexon III under conditions of high alkalinity (pH = = 12-13). Fluorexon (at pH ≈ 13) is employed as a metal indicator and, if absent, murexide (at pH ≈ 12). The presence of magnesium in the sample does not interfere with titration.

Reagents

1. Potassium hydroxide, a 5 M solution. Dissolve 280 g of anhydrous caustic potash in water, add some more of it, bringing the volume of solution up to 1 litre, and then agitate.

2. Complexon III, a 0.05 M solution. The titre of the complexon solution with reference to calcium oxide (T_{CaO}) is computed according to the formula

$$T_{CaO} = D_c \times 56.08 \frac{1}{1000} = D_c \times 0.5608 \text{ g/ml}$$

where D_c is the molarity of the complexon III solution fixed relative to the standard zinc solution; 56.08 is the relative molecular mass of calcium oxide.

3. Indicator — malachite green, a 0.2 % alcoholic solution.

4. Indicator — fluorexon, a dry mixture with potassium chloride in the ratio 1 : 100, or else indicator — murexide, a dry mixture with potassium chloride in the ratio 1 : 100.

The assay procedure. Upon separation of hydroxides take an aliquot part of 100 ml from the filtrate with a pipette, transfer it into a tapered flask of 500 ml capacity, add 100 ml of distilled water and one-two drops of malachite green. The solution then assumes a light-turquoise colouration. Thereupon add a 5 M caustic potash solution in small lots by stirring continuously until discolouration (10-12 ml), following which add again an excess of caustic potash in an amount of 10 ml. To the resulting strongly alkaline solution with pH ≈ 13 add 30-50 mg of fluorexon in the form of a dry indicator mixture with potassium chloride; the solution assumes then a yellowish colour with an intensive green fluorescence. The solution thus coloured is to be titrated with a 0.05 *M* complexon solution till its colour changes to golden-orange with a sharply decreased green fluorescense. The solution is prepared immediately before titration of the sample.

In the absence of fluorexon the titration may proceed by using murexide as an indicator under similar conditions, but with pH $\approx$ 12. Then the colouration of the solution changes from pink to amethystine.

The CaO content (in %) is found from the formula

$$\mathrm{CaO} = \frac{T_{\mathrm{CaO}} V \times 500}{100a} \times 100 \qquad (36)$$

where T_{CaO} is the titre of the complexon III solution in grams of calcium oxide per 1 ml; 500 is the total volume of the test solution in ml; 100 is the volume of the aliquot part of the solution, taken for determination.

Determination of magnesium oxide. Complexonometrically, magnesium is determined in an alkaline medium within a pH interval of 8 to 11. Chromogen black (with pH $\approx$ 9), or beryllon (with pH $\approx$ 11) are used as metal indicators in titration.

Since calcium is also titrated in the said conditions, the magnesium content is computed as a difference after calcium has been titrated.

Reagents

1. Buffer solution with pH $\approx$ 11. Dissolve 20 g ammonium chloride and 10 g of caustic potash or 7 g of sodium hydroxide in distilled water, add 400 ml of a 25 % ammonia and dilute with water to a volume of 1 litre.

2. Complexon III, a 0.05 *M* solution.

The titre of the complexon solution is estimated with relation to magnesium oxide (T_{MgO}) according to the formula

$$T_{\mathrm{MgO}} = D_c \times 40.32 \frac{1}{1000} = D_c \times 0.04032 \text{ g/ml}$$

where D_c is the molarity of the complexon III solution fixed with reference to a standard zinc solution; 40.32 is the relative molecular mass of magnesium oxide.

3. Indicator — chromogen black ET-00, a dry mixture of the indicator with potassium chloride at a ratio of 1 : 100, or indicator — beryllon II, dry mixture of the indicator with potassium chloride in the ratio 1 : 100.

The assay procedure. On separation of hydroxide take an aliquot part of 100 ml with a pipette from the filtrate and transfer it into a 500 ml tapered flask, add 100 ml of distilled water, 10-12 ml of the buffer solution and about 30 ml of a dry indicator mixture. Titrate the resulting solution with a 0.05 M complexon III solution for as long as on application of the chromogen black ET-00 the colouration changes from violet to a pure blue and, on application of beryllon II, from blue to amethystine.

The MgO content (in %) is found from the formula

$$MgO = \frac{T_{MgO}(A - B) \times 500}{100a} \times 100 \qquad (37)$$

where T_{MgO} is the titre of the complexon III solution, expressed in g of MgO per ml; A is the quantity of the complexon III solution spent for titration in a 100 ml aliquot part of the sum-total of calcium and magnesium, in ml; B is the quantity of the complexon III solution in ml spent for titrating calcium in 100 ml of the test solution.

Determination of SO_3

Reagents

1. Hydrochloric acid, density 1.17.
2. Barium chloride, 5 % solution.

The assay procedure. On separation of hydroxides take a 200 ml aliquot part with a pipette and transfer it into a 400 ml cup, heat up to the boiling point, acidate with hydrochloric acid till the yellow colouration of the solution changes to pink (in precipitating sesquioxides — Al_2O_3, Fe_2O_3 — use is made of the methyl red indicator, which is pink in the acid medium and yellow in the alkaline one). To the boiling solution pour 20 ml of a boiling barium chloride solution. Boil the solution with the residue for 10 min, set aside for 4-5 hours (and even longer) and then pass through a dense double filter (with a white tape). Wash off the deposit on the filter with cold distilled water for as long as the reaction for the chlorine ion disappears. Transfer the residue together with the filter into a preliminarily calcined and weighed porcelain crucible, slightly dry on a range, incinerate and calcine in a muffle for 20-30 min at a temperature of 800 °C.

Cool the crucible in an exsiccator and weigh it. Repeat calcination till constant mass.

The sulfur trioxide (SO_3) content (in %) is calculated according to the formula

$$SO_3 = \frac{v \times 0.343 \times 500}{200a} \times 100 \qquad (38)$$

where v is the mass of the calcined residue, g; a is the weighted sample lot of cement, g; 0.343 is the coefficient of the barium sulfate conversion to sulfur dioxide; 500 is the total volume of the solution, in ml; 200 is the volume of an aliquot part of the solution taken for determination.

Determining the sum-total of the alkaline metal oxides. This is a labour-consuming operation and its performance requires a particular thoroughness.

Reagents

1. 8-oxyxynoline-ortho, a 1.5 % solution.

Dissolve 1.5 g of oxyxynoline in 8-10 ml of hydrochloric acid (1 : 3) and adjust with water to 100 ml.

2. Sulfuric acid, specific weight 1.84.
3. Hydrofluoric acid, a 40 % solution.
4. Hydrous ammonia, a 25 % solution.
5. Ammonium oxalate, a 4 % solution.
6. Hydrochloric acid, solution 1 : 3.

The assay procedure. Wet 0.5 g of cement with a few drops of water in a platinum dish, add 2 ml of concentrated sulfuric acid and 10 ml of hydrofluoric acid.

Boil down dry the dish contents on a water bath, then transfer the dish onto a sand bath and heat until disappearance of the sulfuric anhydride fumes. Thereafter, transfer the dish into a muffle and calcine at 600 °C for 10 min. Cool the dish, treat its contents with 30-40 ml of hot water, add 4 ml of a 25 % ammonia solution, agitate and pass through a loose filter into a 200 ml cup. Wash the residue 12 times with small lots of warm water. Reject the deposit. Heat the filtrate to the boiling point, add 10 ml of a 4 % ammonium oxalate solution. Filter off the precipitate through a dense double ashless filter and wash it several times with warm water. Reject the deposit. The filtrate is to be evaporated so as to obtain a volume of 10-15 ml.

To precipitate aluminium and magnesium left in the solution add 2 ml of a 25 % ammonia and 5-7 ml of a 1.5 % oxyxynoline solution till the solution above the precipitate turns yellow. Cool the solution and let it pass through a dense filter into a porcelain dish. Wash the deposit 6-7 times with warm water, boil down dry the filtrate and calcine it at 600 °C to remove ammonium salts.

Dissolve the resultant residue in the porcelain dish with hot water and filtrate into a weighed platinum dish, wash 7-8 times with water and add two-three drops of concentrated sulfuric acid to the filtrate.

Boil down dry the contents of the dish on a water bath, transfer the dish onto a sand bath and heat it for as long as the sulfuric anhydride fumes disappear. Thereupon, transfer the dish into a muffle, calcine for 10 minutes at 750-800 °C and weigh.

Dissolve the resulting alkaline metal sulfates in a small quantity of cold water. Should there remain an insoluble residue, filtrate it off and wash with warm water.

Transfer the filtrate with the deposit into the dish in which the alkali were evaporated, incinerate the filter, calcine the residue at 750-800 °C and weigh.

The content of the sum-total of the alkaline metal oxides $Na_2O + K_2O$ in per cent is calculated by using the formula

$$Na_2O + K_2O = \frac{(v_1 - v_2) \times 0.488}{a} \times 100 \qquad (39)$$

where v_1 is the mass of the dish with the alkali sulfates, g; v_2 is the mass of the dish with insoluble residue, g; a is the weighed lot of cement, g; 0.488 is the coefficient of conversion of the sum-total of potassium and sodium sulfates into oxides.

Chapter 6

Properties of Cement Slurry and Cement Stone

The properties of cement slurry depend upon a variety of factors, the principal ones include the chemico-mineralogical composition, quality and quantity of aggregates, water-cement ratio, quantity and nature of chemical fillers, mixing temperature, pressure, etc.

The treatment of the plugging cement and its proper sampling have an essential influence on the properties of the slurry and stone. In order that the properties of a sample taken could reflect those of the entire lot it is necessary for the sampling to be made in an adequate fashion. Before testing, each sample is to be screened through a No. 09 sieve (with its meshes measuring 0.9 × 0.9 mm in clear).

The entire technology of the oil-well cementing and, in a large measure, proper separation (sealing off) of oil-bearing beds depend on the properties of the plugging mixture and its composition. The quality of the well casing is largely determined by the properties of cement slurry and cement stone: sedimentation stability of the slurry, its water (filter) loss, mobility (fluidity), changes in structural strength with time, setting and thickening times, and density, and the mechanical strength of cement stone, its permeability, and volumetric changes.

The properties of cement slurries and cement stone can be modified by introducing into them aggregates and active additives and also through their treatment with chemical reagents.

6.1. Sedimentation Stability of Cement Slurries

During the first minutes (sometimes hours) the cement grains in the cement slurry remain separated and there occurs water loss, *viz.* the mass of cement moves downward and the water rises. The large and small cement grains fall down practically at the same speed. The water, while passing through the descending mass of cement, remains nearly pure, free of minute particles.

The distribution of water in the settled bulk of cement is not the same everywhere. In the bottom part of a vessel filled with cement slurry the quantity of water in the mass of cement is smaller than in the upper part of the vessel. The mass of the cement slurry over the height of the vessel is heterogeneous.

The maximal quantity of water that can be released from a cement slurry as a result of sedimentation of cement particles is known as the yield of water of the cement slurry. It depends upon the nature of the cement, its chemical treatment, water-cement ratio, and the dispersion of cement flour.

Water separation leads to the formation of water-rich zones, changeability of the cement slurry and stone properties in the annular space of the well.

The separation of water from cement slurry can be reduced by introducing into it structure-forming materials (bentonite, some chemicals), increasing the specific surface of the cement and bringing down the water-cement ratio.

6.2. Water Loss of Cement Slurry

The process of water separation occurring in cement slurry under the effect of a pressure differential is known by the name of water loss. The water loss rate is largely contingent upon the adopted water-cement ratio; it is inversely proportional to the square of the specific surface, fineness of the cement and the quality of the aggregate. The water loss rate is also inversely proportional to the viscosity of the liquid phase of the slurry.

Plugging mixtures for cementing wells, the water loss of which closely approaches zero, should be considered as best. Then, many possible complications are eliminated and a probable soiling of the bottom-hole area of the producing formation with the mixture filtrate is prevented.

At a constant temperature and the pressure differential increasing up to 5 MPa, the water loss of cement slurry goes up. The temperature influences the water loss rate, changing the viscosity of water and the hydration rate. Then, the initial water loss rate must increase with mounting temperature. It diminishes thereafter as a result of the growing rate of cement hydration. With the temperature rising from 20 to 250 °C, the water loss rate of the mixtures made of the majority of plugging portland cements usually increases.

The magnitude of cement slurry water loss is greatly dependent on the pressure differential and the level of absolute pressure above and under the filters. In the absence of back pressure, i. e., when the pressure underneath the filters equals the atmospheric one, the water loss with pressure differentials of 10, 15, 20, and 25 MPa continues to be almost invariable. The water loss rate mounts with rising pressure differential and absolute pressure, while with differentials of 30 to 40 MPa, the process of the water loss terminates approximately in 1-2 minutes.

The most effective method of forcing down the water loss of cement slurries is the one involving simultaneous introduction therein of clay and chemical reagents.

Table 19 lists data on the water loss in cement slurries (on introduction of clay), defined by passing them through a paper filter fixed in a standard apparatus, Model BM-6. An addition of clay contributes to lowering the water loss in cement slurries. The results proved better with concurrent introduction of clay and hypan into cement slurries (Table 20). By using the said additives one can practically obtain plugging mixtures with a very insignificant water loss.

Table 19

Water Loss of Cement Slurry with Addition of Clay

Cement slurry		Cement slurry + clay, %											
		4		6		8		10		20		30	
Time, min	Water loss, cm³	Time, min	Water loss, cm³	Time, min	Water loss, cm³	Time, min	Water loss, cm³	Time, min	Water loss, cm³	Time, min	Water loss, cm³	Time, min	Water loss, cm³
0.5	31	—	—	0.5	17	0.5	12	0.5	10	0.5	11	0.5	12
0.7	40	—	—	—	—	—	—	—	—	—	—	—	—
—	—	1	27	1	26	1	18	1	19	1	17	1	16
—	—	2	39	2	30	2	27	2	25	2	28	2	30
—	—	—	—	2.5	39	3	36	3	36	3	34	3	35
—	—	—	—	—	—	3.5	40	3.5	38	3.5	39	3.5	40
—	—	—	—	—	—	—	—	4	40	3.7	40	—	—

The technology of preparation of plugging mixtures with a decreased water loss envisages a uniform mixing of cement and clay with subsequent addition of water containing hypan, preliminarily dissolved therein, in an amount fixed by the laboratory.

The optimal proportion of hypan or carboxymethylcellulose (CMC) is 1.0-1.5 %. Any further increase in the amount of reagents brings with it but an insignificant fall of the water loss. The optimal quantity of clay is 7 to 20 %. The addition of clay has a material influence on the water loss rate, but less so on its absolute magnitude. Along with reducing the water loss by the cement slurries, the reagents introduced therein tend, as a rule, to retard the setting of the plugging mixtures. And should the temperature in the wells not be high enough, the plugging mixtures have to be treated with retarding agents.

Table 20

Water Loss of Cement-Clay Slurry with Addition of Hypan

Cement slurry		Cement slurry + 25% clay + hypan, %									
		0.5		1.0		1.5		1.8		2.0	
Time, min	Water loss, cm^3	Time, min	Water loss, cm^3	Time, min	Water loss, cm^3	Time, min	Water loss, cm^3	Time, min	Water loss, cm^3	Time, min	Water loss, cm^3
0.5	15	0.5	6	0.5	3	0.5	3	0.5	2	0.5	2
1	20	1	9	1	5	1	5	1	3	1	3
3	30	3	20	3	7	3	7	3	5	3	2
—	—	10	26	10	13	10	11	10	7	10	4
—	—	20	41	20	17	20	14	20	7	20	5
—	—	—	—	30	17	30	15	30	8	30	6

6.3. Thickening of Cement Slurry

Thickening of plugging mixtures is determined with a consistometer.

Thickening of cement slurries and its onset depend upon a number of factors, the nature of the cement itself being quite essential. A somewhat increased proportion of tricalcium aluminate in the clinker favours a steeply accelerated thickening of cement slurries.

An increased fineness of cement leads to an accelerated thickening of slurries, while a higher water-cement ratio retards it.

Temperature, pressure and pressure variations exert an important influence on the thickening of cement slurries. With rising temperature the thickening time gets shorter and at about 100 °C it is roughly 15-30 minutes (for slurries prepared from plugging portland cement).

An extension of the thickening time of plugging mixtures can be achieved by using agents retarding the processes of structurization, the quantity and quality of which have to be selected with due regard for concrete conditions prevailing in the well. Among retarders may be counted spent sulfite-alcohol liquor (SAL), CMC, hypan, commercial cream of tartar (CCT), potassium dichromate, Л-6 (hydrolyzed pentosanes) and others.

The higher the pressure, the shorter the thickening time of cement slurries. A pressure of about 100 MPa has a highly marked effect. Thickening of even chemically treated mixtures occurs then over a very short period of time. In such a case one must resort to the use of principally new types of cement (cements with slag as their base, etc.).

When a cement slurry, initially subjected to high temperatures and pressures, experiences sudden, repeated drops in the applied pressure, its thickening accelerates.

6.4. Setting Time of Cement Slurries

In the Soviet practice the suitability of plugging mixtures is determined mostly by the setting time. The setting rate of cement slurries is contingent upon the chemico-mineralogical composition of the cement, its specific surface, water-cement ratio, chemical reagents introduced into the slurry, temperature, pressure and other factors.

All other things being equal, a greater specific surface of the cement and a lower water-cement ratio speed up the setting rate of a cement slurry.

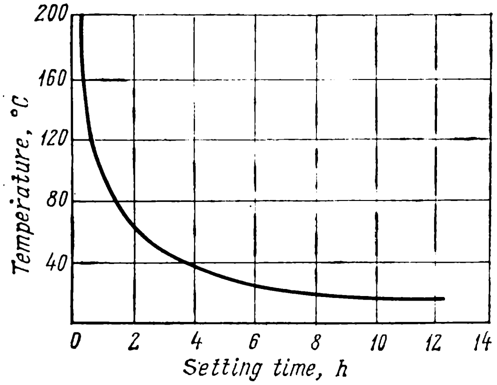

Fig. 48. Setting time of cement slurry as a function of temperature

In the USSR, the study of changes in the physico-mechanical properties of cement slurry and cement stone at elevated temperatures has been commenced earlier than in other countries. It has been found that it is temperature rather than pressure that affects more the rate of the cement slurry setting. Joint action of temperatures and pressures exerts a still greater influence.

Figure 48 illustrates changes in the setting time under the effect of temperature (pressure was maintained at 5 MPa to prevent steam generation in the autoclave). An analysis into the curve changes shows that already at temperatures close to 100 °C setting-time retarders must be introduced into the slurry, for the actual setting time is limited to rather short periods which are obviously insufficient to provide for cementation of a deep high-temperature well.

The effect of pressure alone also tends to cut down the setting time of cement slurries (Fig. 49). With an elevated pressure, starting from the atmospheric and up to 50-60 MPa, the setting time of cement slurries is almost halved. Investigations made in recent years indicate the setting time of cement slurries to become markedly shorter with pressures exceeding 130 to 150 MPa. With rising temperature the acceleration effect due to pressure continues to be quite appreciable.

In the practice of oil-well cementing, when selecting a composition of plugging mixtures at a laboratory, one should never ignore the factor of pressure, and in determining the setting time, one should take into consideration the joint action of temperature and pressure. As the storage period of cements grows longer, their setting takes more time and the toughness increases. The presence in the atmospheric air of moisture, carbon dioxide, etc., is conducive to lowering the activity of the cement and lengthening the setting time. Such a retardation in setting cannot, however, be considered as a method suitable for using in deep high-temperature wells.

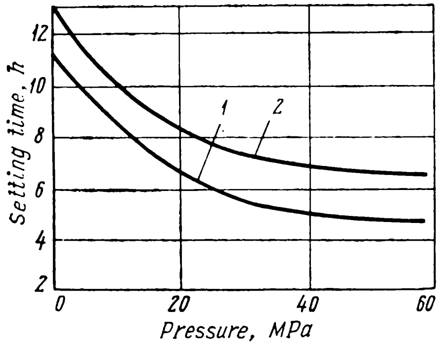

Fig. 49. Setting time of cement slurry as a function of pressure
1 — initial set; 2 — final set

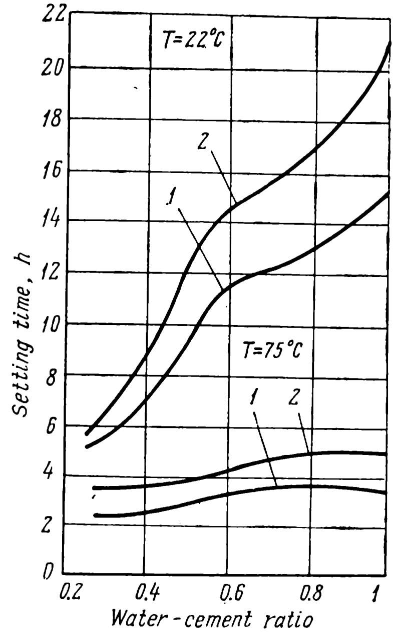

Fig. 50. Setting time of cement slurry as a function of water-cement ratio
1 — initial set; 2 — final set

Figure 50 presents changes in the setting time of cement slurries with variable water-cement ratio occurring under the effect of different temperatures. As noted earlier, at low temperatures a higher water-cement ratio tends to lengthen the setting time of cement slurries, but already at temperatures somewhat above 100 °C the retardation of the setting processes practically cannot be secured by raising the water-cement ratio up to 1.

6.5. Density of Cement Slurry

The most important characteristic of cement slurry is its density. It is practically the sole factor indicative of the quality of a plugging mixture during its preparation and transport to the well. Variations in the density of the mixture point to changes in its other properties, such as pumpability, thickening, strength of stone, etc. For this reason, the control over fluctuation in the density of plugging mixtures should be very strict and such fluctuations must not go beyond fixed limits.

Density fluctuations are often inadmissible on the grounds of technological considerations.

The density of a cement slurry is determined in the main by its water-cement ratio. If a cement slurry does not have to meet special requirements then its water-cement ratio, according to USSR Standard GOST 1581-78, is set at 0.5. The estimated density of portland cement slurry then equals 1.83 g/cm^3. In practice it varies somewhat about this value (1.81 to 1.85 g/cm^3). These variations are explained by the presence of air gaining access into the cement mixture and also by the inconstancy of the density of cement.

Changes in the density values of a cement slurry are influenced by the chemical reagents introduced into it. The greatest influence is exercised by the spent sulfite-alcohol liquor (SAL), which in an amount of more than 0.4 % promotes the formation of foam, entraining therewith air into the slurry during its preparation. But when the slurry gets inside a hydraulic pressure medium, the air bubbles are compressed. The mass of the unit volume of the slurry goes up and this, under definite geological conditions attending sinking of the well, may result in complications.

The same is true with the use of air-containing aggregates (perlite, keramsite and others) for reducing the density of plugging mixtures employed in deep wells.

It is believed that the displacement of clay drilling mud from the annular space of a well by cement slurry is consequent upon the difference between their densities, but it appears impossible so far to establish the range within which this difference varies.

6.6. Intermingling of Mud Fluids and Plugging Mixtures

Intermingling of plugging mixtures with mud fluids is often followed by the formation of thick pastes whose effective viscosity is tens and hundreds of times greater than that of the initial components. Thickening of mixtures during their intermingling leads to a rise of pressure during cementation of wells and in individual cases to complications.

On intermingling of clayey and cement mixtures, thickening does not always follow the same pattern, nor is it equally hazardous. It is determined by the composition of the solid phase of the mixtures and their chemical treatment.

Accordingly, if it comes to the application and choosing of spacer fluids for separating clay muds and cement slurries, one must consider the properties of these fluids and their ability to thicken upon intermingling.

During intermingling of plugging mixtures and mud fluids there are in evidence the following phenomena, common to all of their compositions.

1. During agitation or transport of condensed mixtures of plugging material and mud fluids over pipelines there is observable a continual thinning, whose rate depends upon the composition and chemical treatment of constituents in the mixture. The lowest thinning rate is seen in the case of cement mixtures or mud fluids treated with hypan or CMC.

2. Slag mixtures cause a much less marked condensation of mud fluids than do the portland cement ones. The condensed mixtures that contain slag get thinner quicker than mixtures prepared on the base of the portland cement.

3. Addition of sand, weighting and lightening additives to cements and slags tends to reduce somewhat the condensation. On adding 10-50 % of clay or weighting agents to the cement and bringing the water-cement ratio to the required value (flow — 20-22 cm, for example), the flowability of the mixtures determined by the flow and slump test increases by 10-30 %. A similar effect is produced by an addition of weighting agents or of other nonclayey materials.

4. Addition of graphite to mud fluids has practically no effect on the condensation during their intermingling, while addition of sulphonol and crude oil makes them less thick.

5. Mud fluids are subjected, as a rule, to a complex chemical treatment, so that to take account of and investigate all the possible combinations of reagents is virtually impossible.

To educe the tendency towards condensation of mud fluids on intermingling with plugging mixtures one has to bear in mind that if a mud fluid has been treated with hypan along with other reagents the factor decisive for its properties will be the presence of hypan. With a mud fluid carrying no hypan, but treated with CMC its proneness to condense will be determined by CMC. Should a mud fluid be treated with coal-alkali agent (CAA) and lignosulfonates, its properties will be determined by CAA. Potassium dichromate does not affect substantially the thickening during intermingling of mixtures.

When employing petroleum emulsion, oil asphalt mixtures, or those with petroleum as their base, the stabilizers carry no hypan or CMC and in this case no condensation of the mud fluid and cement mixture is observable. On the other hand, the treatment of mud fluid based on petroleum with hypan or CMC leads to its thickening when intermingling with a cement slurry. If a mud fluid is treated with several reagents, then these are arrayed in an order of hypan > CMC > CAA > lignosulfonates, as regards the influence they have on the condensation factor. The action of reagents on the properties of mud fluids depends, however, not only on the kind but also on the quantity of the reagents added to the mud fluid. In some instances, when, for example, the quantity of CAA added to the fluid is scores of times as great as that of hypan or CMC, the effect of the latter additives on the condensation during intermingling may be brought to naught.

6.7. Contraction Effect in Hydration of Cement and in Hardening of Cement Slurry

An important property of the cement slurry is contraction by which is understood to be shrinking (reduction) of the summary volume of the cement and water in the course of the hydration reaction. Distinction is made between the physical and chemical contraction. In hardening of plugging mixtures both types of contraction are in evidence, but the scope of the total effect is largely due to chemical processes.

In the course of hardening of hydraulic binders the overall volume of the cement-water system diminishes (after V. V. Nekrasov), mainly because of a different density of initial and final products. This is conditioned by the readjustment of the crystal lattices in the original clinker minerals during their hydration, which changes their atomic pattern to the molecular one. In this connection, the new term "tightening" given to this phenomenon by S. D. Okorokov goes deeper to the heart of its nature, whereas the designation "contraction" corresponds rather to the external manifestation of the process.

Outwardly, the contraction finds its expression in absorption of water (or gas) that is in contact with a cement slurry undergoing hardening. Thus, if water is poured over and above a freshly prepared cement slurry placed in a vessel, it will be getting absorbed parallel with hardening of the slurry. With the environmental temperature going up, the water absorption rate increases. The absorption of water will discontinue after complete hydration of the cement grains. Once the full hydration of the portland cement comes to an end, the quantity of absorbed water (i. e. contraction) amounts to 7-9 ml per 100 g. The contraction in highly active cements, when these combine with water (50 %), reaches in 28 days of hardening under ordinary conditions 50-65 % of the maximum, its further manifestations slowing down greatly. Little-active cements (of low grades) display by this time a degree of contraction that equals 30-40 per cent of the maximum.

When a plugging mixture hardens in a hermetically sealed vessel the air in the latter becomes rarefied, i. e. the pressure therein falls, which can be demonstrated with a simple apparatus shown in Fig. 51. This consists of a flask with a cement mixture poured into it and a mercury or water pressure gauge.

Figure 52 presents curves featuring absorption of water by cement slurries at different temperatures.

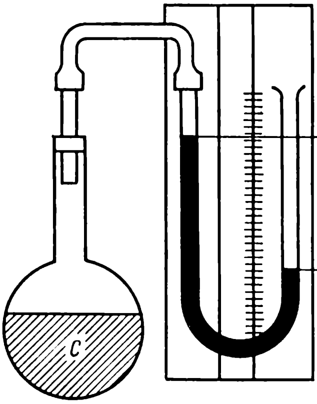

Fig. 51. Schematic diagram of apparatus for determining the contraction of cement slurries

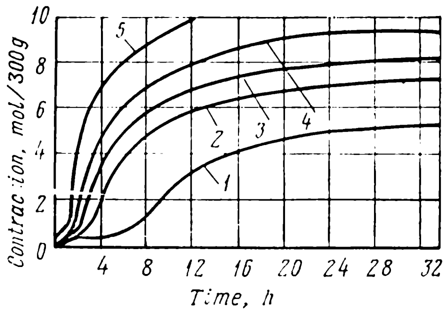

Fig. 52. Absorption of water by cement slurry and stone during hardening at various temperatures

1 — *t* = 22°C; *2* — *t* = 50°C; *3* — *t* = 80°C; *4* — *t* = 100°C; *5* — *t* = 125°C

In hardening of cement slurries the drilling (mud) fluids that come into contact with the former become dehydrated, fissured and porous.

The contraction phenomenon is of prime importance for the formation of a cement ring in the annular space and explanation of the emergence of channels therein.

6.8. Mechanical Strength of Cement Stone

The mechanical strength of cement stone is dependent upon a number of factors, the principal of which are the chemical and mineralogical compositions of cement, water-cement ratio, specific surface of cement, presence of aggregates and chemical additives, and conditions of hardening, chiefly the temperature. The relationship between the mechanical strength of portland cement stone and the chemical and mineralogical compositions of the cement begins to conspicuously manifest itself with long periods of hardening. The same factor appears to be decisive in the formation of the gel and crystal phases in cement stone and in their correlation. At the inception of hardening the strength gain is achieved at the expense of alite. The strength is determined in a great measure by the rate and extent of the stone hydration. At later dates, the strength of cement stone is secured by the presence of the belite constituent in cement.

The mechanical strength of cement stone grows faster in the case of high specific surface, for then the reaction of hydrolysis and hydration proceeds at a quicker pace. But one cannot boundlessly increase the specific surface of cement stone, as this would require a greater quantity of water of plasticity for mixing and increase the cost of cement. For work at high temperatures and pressures it is preferable to use portland cements of not too high specific surface, but rather of a definite granulometric composition (grain size). The specific surface of plugging portland cements lies in the range 2000 to 4000 cm²/g.

Cement setting accelerators help raise in most cases the initial mechanical strength, but as the period of hardening gets longer the strength of the cement stone usually diminishes somewhat earlier than in untreated specimens. Setting retarders tend to lower the strength of the cement stone in the early period of hardening.

Depending upon the conditions of hardening, additives can produce either a favourable or a negative effect on the mechanical strength of the portland cement stone.

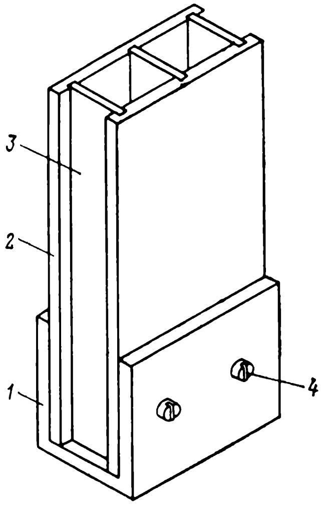

Fig. 53. Mould for testing cement slurries for hardening in autoclaves under high temperatures and pressures

1 — clamp; *2* — web; *3* — side plate; *4* — clamping screw

Ordinarily, aggregates carrying 80-95 % of silica (quartz sand, opoka, etc.) are not apt to raise the initial strength of the cement stone, but with its ageing the strength increases. At temperatures in excess of 110-120°C the aforementioned aggregates turn into active materials and thus increase the mechanical strength of cement stone.

Argillaceous materials and their mixtures with water practically always bring down the mechanical strength of the stone. This phenomenon is seen to occur when cement is mixed with dry bentonite powder, in mixing cement with mud fluid and in intermingling of a cement slurry with a mud fluid while they are moving in the annular space of a well. An exception are cases when small amounts of a powder-like clay are introduced into a cement slurry (or cement), for the former tends to lower the water-cement ratio.

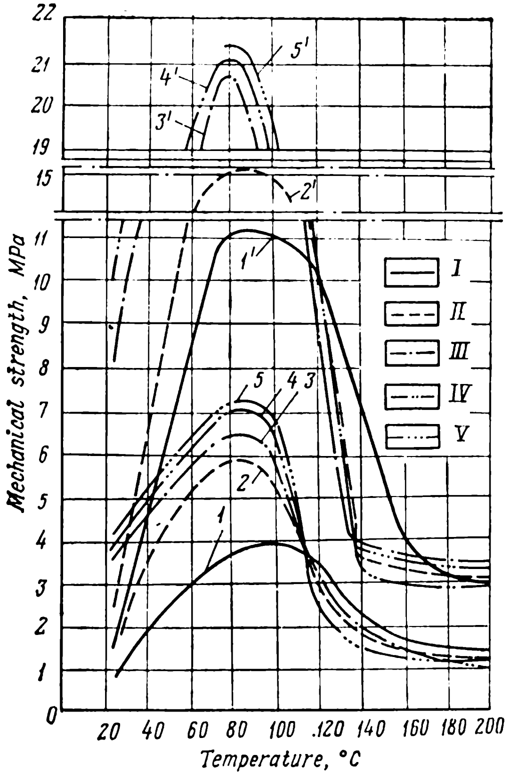

Fig. 54. Mechanical strength of cement stone as a function of temperature
Age of specimens: *I* — 12 h; *II* — 12 h; *III* — 48 h; *IV* — 96 h; *V* — 168 h. Strength: *1* through *5* — in bending; *1'* through *5'* — in compression

Test procedures. The testing of cement specimens at temperatures of 22 and 75 °*C* is made according to the pertinent standard. At elevated temperatures and pressures the testing procedure differs somewhat from the standard one. The cement slurry is poured into collapsible moulds (Fig. 53) oiled with machine grease and placed in an autoclave. The cement is mixed at once with a needed quantity of water. With the hermetically sealed autoclave the rate of the temperature rise should be 2-5 °C per minute. After 48 ± 2 hours of curing, the specimens are withdrawn from the autoclave. The physico-mechanical properties of the cement stone are influenced in a definite fashion by the cooling rate of the autoclave. The latter is to be cooled for approximately 1 hour. On their removal, the cement specimens should also be cooled for 1 hour.

The effect of temperature (Fig. 54) *and pressure on the mechanical strength of cement stone.* The temperature and pressure have quite an important effect on the strength of plugging portland cement stone. At 22 °C the strength of the stone increases with longer periods of hardening, the pressure playing a vital role, for with its rise the strength of the specimens becomes greater.

At 75 °C and a longer hardening time the stone also gains in strength, but as soon as the third-fourth day it becomes stable, while after one-two months of hardening its strength starts declining. The pressure causes some changes, namely, the early strength increases while that of the later date remains unchanged.

At 110 °C the mechanical strength of the cement stone with longer periods of hardening increases, but its growth rate and absolute strength values decline. Pressure has virtually no effect on the change in the mechanical strength of the portland cement stone.

At temperatures of 140 and 200 °C the portland cement stone strength is not high and there is a tendency towards its decrease as the time of hardening draws in length. Pressure either fails to materially downgrade the strength, or else manifestly contributes to its fall.

6.9. Permeability of Cement Stone

The permeability of cement stone is one of its main characteristics. It depends on a number of factors among which the most important are the nature of cement and aggregates, water-cement ratio, ambient conditions (temperature, pressure) and hardening time. Essential is also the chemical composition of water contacting the hardening and already hardened cement slurry.

Figures 55 through 57 show changes in the permeability of cement stone which undergoes hardening under different conditions and during diverse time periods. At 22 °C its perviousness decreases with prolonged hardening, while at 75 °C it is insignificant and varies within a narrow range. At 110 °C and a pressure of 30 MPa the rate of the strength changes is slower, while permeability amounts to (1.5 to 2.5)× 10^{-3} μ^2. At higher temperatures the permeability of portland cement stone rises steeply.

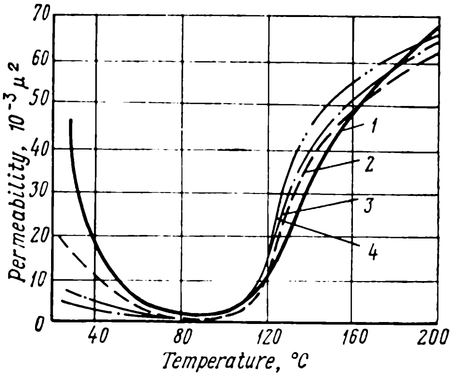

Fig. 55. Permeability of cement stone as a function of temperature

Age of specimens: 1 — 12 h; 2 — 24 h; 3 — 48 h; 4 — 96 h

In practice, the water-cement ratio of slurries may vary widely, and this ratio has a great bearing upon changes in the permeability of cement stone. Its increase brings with it a greater volume and number of capillary pores. In the course of hydration they get filled with the gel constituent whose permeability is quite low. Curves presented in Fig. 57 indicate that after two days of hardening at 130 °C and under a pressure of 40 MPa the permeability of cement stone is somewhat greater than 2×10^{-3} μ^2 when the water-cement ratio amounts to 0.5. With the temperature going up to 150 and 170 °C, the permeability increases to 25.5×10^{-3} and 32.3×10^{-3} μ^2, respectively. When the water-cement ratio is 0.6 to 0.7 and temperature 150 to 170 °C, the permeability rises up to (60 to 80)$\times 10^{-3}$ μ^2, which points to the need of a strict control over the changes in density. At higher temperatures and a considerable proportion of water in the mixture (80 to 90 %) the permeability of stone is as high as (100 to 150)$\times 10^{-3}$ μ^2.

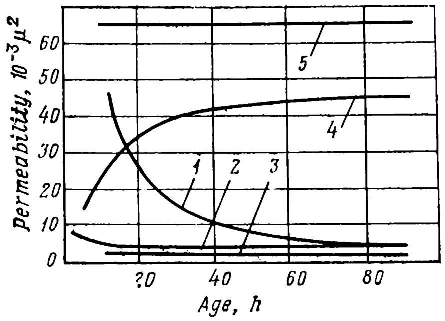

Fig. 56. Permeability of cement stone as a function of its ageing at various temperatures and pressures

1 — $t = 20$°C; 2 — $t = 75$°C; 3 — $t = 110$°C; $p = 30$ MPa; 4 — $t = 140$°C; $p = 40$ MPa; 5 — $t = 200$°C; $p = 50$ MPa

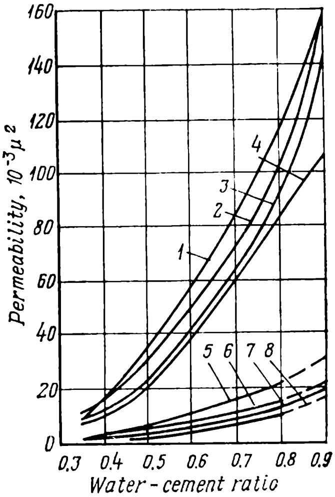

Fig. 57. Permeability of cement stone as a function of water-cement ratio

Age of specimens and their ageing conditions: 1 — 2 days at $t = 170$°C and $p = 40$ MPa; 2 — 2 to 7 days at $t = 170$°C and $p = 40$ MPa; 3 — 7 days at $t = 150$°C and $p = 40$ MPa; 4 — 2 days at $t = 150$°C and $p = 40$ MPa; 5 — 7 days at $t = 130$°C and $p = 40$ MPa; 6 — 2 days at $t = 22$°C and $p = 0.1$ MPa; 7 — 2 days at $t = 130$° and $p = 40$ MPa; 8 — 2 days at $t = 75$°C and $p = 0.1$ MPa

The most effective way of reducing the permeability of portland cement stone at high temperatures and pressures is introduction of quartz sand into the slurries.

6.10. Adhesion of Cement Stone to Casing String Metal and to Rocks

Adhesion of the hardening cement stone to casing pipes and rocks is determined by numerous factors, the principal ones are the cleanliness and roughness of the contacting surfaces, the nature of the cement, the temperature of the hardening medium and others. Should the surface of the casing pipes and of the rock be coated with a mud fluid, no bond between the pipes and the cement mixture and stone can be expected.

The nature of cement stone adhesion to metal finds its explanation in the formation of an intermetallic layer with a definitely arranged crystal lattice. This layer makes part of both the metal and the cement stone. Its strength depends on the bond between the cement stone and the metal, the nature of the stone and on other factors. Of no small importance in this connection are the frictional forces that develop with squeezing of the metal by the stone. With special reference to conditions attending the performance of the cement ring in a well, by adhesion is meant a concurrent action of forces that hold in place a rod by means of a cement stone ring.

Figure 58 presents in coordinates *Q-Z* (loading *vs* dislocation of a rod) a typical diagram illustrating the pull-out of a metal rod from the cement stone held in a casing. First, the load on the rod rises to the maximum not attended by displacement of the rod; then, once the bond between the cement stone and the metal has been disrupted, the load drops down to a certain value and remains roughly constant till the rod projects out of the cement stone over a considerable height.

At 22 °C and under atmospheric pressure the bond between the hardening cement stone and the steel rod sharply gains in intensity (Fig. 59) over a lengthy period. At 75 °C a considerably increased adhesion is observed during the first 12 hours with a tendency towards stabilization by the seventh day. With the temperature going up to 110 °C and the pressure to 40 MPa and within a broad range of time-periods, the adhesion of cement stone to the metal (steel) rod remains virtually constant. When the temperature goes up from 140 to 200 °C under constant pressure the adhesion drops and then continues to be roughly constant.

If the surface of a steel rod is covered with a mud fluid, treated and untreated alike, the adhesion declines in all instances down to zero. High temperature and pressure produce no upward effect on the adhesion of a hardening cement slurry to a mud-coated steel rod.

The bond between the cement stone and a rod with local lugs is determined by the load transmitted to the stone by the area of the lug on the rod.

In securing proper contact of the cement slurry (stone) with rocks the adhesion is dependent upon the mineralogical composition of the cement and the rocks (particularly upon the presence of an argillaceous material) and conditions attending hardening (temperature and pressure), as well as on the state of the rock surface and the degree of its saturation with water.

In the actual field conditions the walls of a well are covered with a muddy crust and a drilling fluid. To clear the walls so as to secure the physico-chemical processes over the contact surfaces of the rock and cement stone to run their proper course is impossible so far.

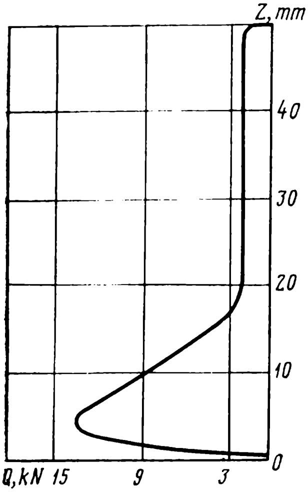

Fig. 58. *Q-Z* (load — displacement) diagram for the case of pullout of a metal rod from cement stone held in a casing

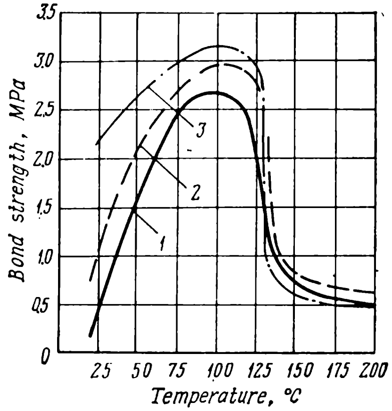

Fig. 59. Strength of bond between cement stone and a steel rod as a function of temperature

Age of specimens: *1* — 12 h; *2* — 24 h; *3* — 96 h

Elementary estimates show that a 10 metre long cement stone ring with an adhesion of 0.01 to 0.02 MPa can easily hold in place 3000 m of a 168-mm dia casing string (with a per-meter mass of 50 kg) even in the absence of any couplings and projections on the casing, and without strict observance of its vertical position. In the field practice this is confirmed by clasping in place (tacking) of filling (charging) pipes in re-cementing jobs, when the cement mixture in the well just begins setting and its strength is low as yet.

6.11. Changes in Volume of Plugging Cements (Slurries and Stone)

Under the effect of different factors cement stone can become subject to shrinkage or expansion. Several types of shrinkage are known.

Physical shrinkage is caused, above all, by evaporation of certain quantity of water from slurry and stone in the process of hardening. It depends on the specific surface of the cement, its mineralogical composition, the water-cement ratio, the presence and physical properties of the aggregate, the temperature and conditions of evaporation. The shrinkage of cement stone begins only after the whole of free water has been removed from it.

Chemical shrinkage is conditioned by the fact that the hydrates which form when the portland cement is being mixed with water occupy a smaller volume than does the sum-total of the volumes of water and anhydrous compounds. It can be reduced by a proper selection of the mineralogical composition of the clinker, by introduction of aggregates, etc.

Thermal shrinkage is due to a gradual cooling of the cement stone with a slower rate of heat evolution.

Hardening of the cement stone in atmospheric evironment often results in reduction of its volume, in shrinkage. Aqueous medium promotes expansion of the cement stone, which is kept down when the mixture is made to contain an elevated proportion of inert aggregates. All other conditions being equal, the swelling of the stone in water is less marked than its shrinkage in the air. Under favourable conditions the volume of the cement slurry starts increasing even before it has become completely set. A protracted expansion of the cement stone in hardening is explained by the pushing action of the developing hydration products.

Many years of observation over the hardening of the cement slurry and stone in autoclaves at high temperature and pressure indicate that the withdrawal of specimens from moulds greased with machine oil and kept in an autoclave presents some difficulties, this bearing proof to the expansion of the cement stone.

While the cement slurry and stone undergo hardening in hermetically sealed autoclaves a pressure drop of up to 8-10 % of the specimen volume is observed, which is suggestive of the cement stone shrinkage. An essential part here plays the contraction of the stone as a result of which some amount of water in the autoclave is absorbed by the specimen. This does not mean, however, that the overall dimensions of the stone have diminished — they can even become larger.

Laboratory Work No. 3

I. *The sedimentation stability of a cement slurry may be determined as follows.*

Pour a freshly prepared cement slurry into a graduated cylinder by filling it to $^3/_4$ of the height. Allow the slurry to stay. Water emerges above the cement slurry. Measure every 5 minutes the height of the cement slurry. To speed up the work, it is recommended that experiments should be conducted with slurries having an elevated water-cement ratio.

II. *Determination of the contraction effect in cement slurries undergoing hardening.*

Pour a cement slurry into a graduated cylinder (which may be replaced by a glass cup with a strip of graph paper pasted onto it) so as to fill it up to $^2/_3$ of the height. Pour cautiously water on top of the slurry to fill the remaining $^1/_3$ of the cylinder. Seal the cylinder hermetically to avert evaporation of water.

Record every hour the graduation line at which lies the concave part of the meniscus. By using the results of measurements plot a curve of water absorption by the hardening cement slurry (stone).

To speed up the process of water absorption, place the cylinder with the cement slurry into a water bath or a thermostat and register the contraction effect in the cement mixture (stone) at a certain temperature (commonly at 75 °C).

III. *Determination of the water loss of cement slurry.*

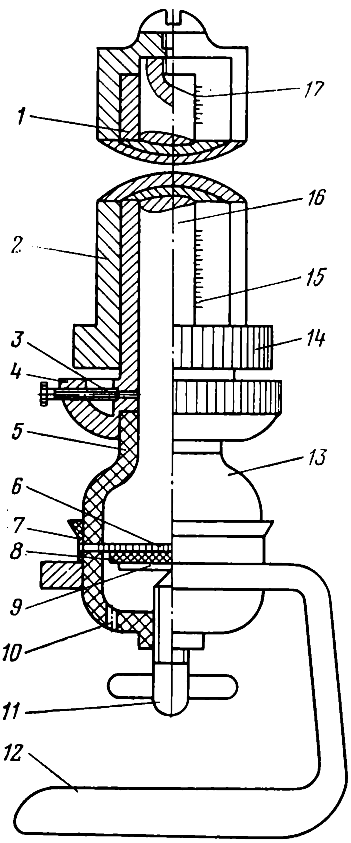

Fig. 60. BM-6 Model apparatus

1 — barrel; *2* — outer cylinder; *3* — bleeding valve needle; *4* — oil pan; *5* — filter bowl; *6* — filter paper; *7* — perforated plate; *8* — seal; *9* — shut-off valve disc; *10* — water drain hole; *11* — shut-off valve screw; *12* — bracket; *13* — filter bowl top cover; *14* — weight ring; *15* — scale; *16* — plunger; *17* — reference line

The water loss of cement slurry may be measured with the help of the BM-6 Model apparatus. The latter (Fig. 60) is intended to measure the water loss of mud fluids under a pressure of 0.1 MPa.

To determine the water loss, withdraw the plunger from the pressure cylinder, unscrew the filter bowl and the pan from the outer cylinder and remove the perforated plate and the valve. Put two sheets of wetted filter paper over the perforated plate and place the latter into the bowl with the paper turned inside. A pin in the bowl's recess should then go into the groove of the perforated plate. Unscrew the valve in the pan and screw on the pan. Insert the bowl into a slot (ring) of the bracket. Tighten by hand the threaded connection between the bowl and the pan. Screw on the shut-off valve of the pan. Fill the bowl with cement slurry through the neck by keeping the level to lie 3-4 mm below the brim of the neck. Screw the pressure cylinder on the neck. Pour a low-viscosity machine oil into the cylinder along its inner wall by keeping the oil in the cylinder at a level 1 cm below the top edge of the liner. Insert the plunger with the scale. By unscrewing the obturating needle let the plunger go down till zero reading of the scale is in line with the mark on the top edge of the cylinder's liner. Open the valve by giving the screw a half-turn and start the timer. To rule out the friction effect give the plunger a few turns by hand. After a lapse of a certain period of time (usually 30 minutes) the water loss is found from the reading opposite which the graduation line has stopped. The water loss of cement slurry not subjected to a special treatment habitually terminates much earlier. This is then said to be the maximum water loss over a definite period of time.

The apparatus is taken apart, the obturating needle opened, the oil drained and all parts cleared from the cement slurry.

IV. *Determining the density of cement slurry.*

The density of cement slurry is determined with areometers (Fig. 61), Models АГ-1 and АГ-2. The АГ-1 Model is intended for determining the density of slurries from 1 to 2.5 g/cm^3 and the model АГ-2, to measure density from 0.9 to 2.4 g/cm^3. The areometer is made up of float *1* with a specific weight scale at its top, bowl *2* and removable weight *3*. If the scale 1-1.8 g/cm^3 is used for the read-out, the device is employed with the weight and in case of the 1.7-2.5 g/cm^3 scale, without it. The handling of the areometer is simple. Bowl *2* is filled with cement slurry to the brim and is secured to the float by letting the pins of the float enter its slots. The excess slurry escapes through the openings. The cement slurry on the surface of the bowl is washed away and the areometer is immersed into a pail with water. The graduation line on the float scale is read out at the water level and this indicates the density of the cement slurry in the bowl.

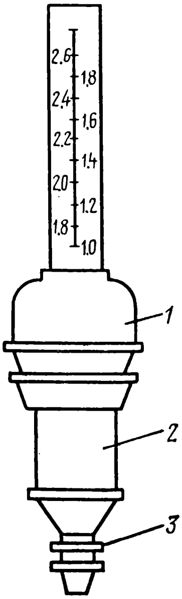

Fig. 61. Areometer for determining the density of plugging mixtures

V. *Determining the thickening of drilling fluids upon their intermingling with plugging mixtures.*

Mix 600 g of plugging cement (or slag) with chemically treated water.

Mix 180 cm^3 of drilling mud with 20 cm^3 of cement slurry and stir by hand until the thickness of the resultant mixture reaches its maximum, which usually takes from one to three minutes. Then find the fluidity (spreadability) of the mixture with the aid of a standard fluidity test cone.

Following the same procedure, measure the fluidity of mixtures containing 100 cm^3 of drilling mud and 100 cm^3 of cement slurry and 20 cm^3 of mud and 180 cm^3 of slurry. The initial fluidity of both the drilling mud and the cement slurry must be at least 20 cm. Using the experimental results obtained, plot the fluidity (in cm) of the mixture as a function of the drilling mud and cement slurry proportions in it.

VI. *Following the procedures described in this chapter, determine the initial and final setting times of cement slurries and the mechanical strength of cement stone.*

Chapter 7

Plugging Cement

In this country, plugging portland cement, i. e., portland cement specially intended for cementing oil and gas wells, is manufactured in accordance with the pertinent USSR standard.

The standard consists of several individual sections dealing with the definition, specifications, acceptance, testing, transportation, and storage of the cement.

7.1. Definition and Composition of Plugging Cement

Depending upon its purpose, plugging portland cement is put out in two types, *viz.*, for "cold" and "hot" wells.

Plugging portland cement is a variety of portland cement produced by way of a joint fine grinding of clinker and gypsum in an amount needed to control setting and hardening times.

Introduction of mineral additives to the clinker at the time of its grinding is admissible, *viz.*:

for "cold" wells — active mineral additives in an amount of not more than 15 % of the mass of the finished product, or inert additives in an amount not exceeding 10 %, in the form of quartz sand or crystalline limestone;

for "hot" wells — active mineral additives of sedimentary origin only, or granulated blast-furnace slag in an amount of not more than 15 %, or quartz sand in an amount not exceeding 10 %.

Granulated blast-furnace slag should meet the requirements specified in the pertinent USSR standard.

7.2. Specifications for Granulated Coke-Smelting Blast-Furnace Slags

Depending upon their chemical composition, granulated blast-furnace slags are classified into two kinds, *viz.*, basic and acid. Basic slags are subdivided into three grades — 1st, 2nd and 3d, and acid ones into two grades — 1st and 2nd. Acid slags of the second grade are broken up into two separate groups, namely, 1st and 2nd.

As to their chemical composition, the granulated blast-furnace slags must meet the following requirements (Table 21).

Table 21

Characteristics	Basic slags			Acid slags		
					2nd grade	
	1st grade	2nd grade	3d grade	1st grade	1st group	2nd group
Modulus of basicity $(\%\ CaO + \%\ MgO)/(\%SiO_2 + \%Al_2O_3)$, not less than	1.0	1.0	1.0	0.9	0.7	0.6
Modulus of activity $(\%\ Al_2O_3)/(\%\ SiO_2)$, not less than	0.25	0.20	0.12	0.40	0.30	0.50
Manganous oxide content (MnO), not more than	0.2	4.0	3.0	2.0	4.0	2.0

Acid slags of the 2nd grade are defined by the factors characterizing one of the groups, the slags of both groups being equivalent.

By mutual agreement between the supplier and consumer a lower modulus of basicity is admitted for basic slags of the 2nd and 3d grades, which, however, should not be lower than 0.95.

The moisture content in the slags is not standardized, but is indicated in the certificate. Slags obtained in smelting of different kinds of pig iron (foundry, conversion, etc.) should not be intermingled during their granulation, storage and delivery. The quantity of lumpy slag (escaping granulation) in a lot of slag should not be greater than 5 % by weight. The size of such lumps must not exceed 100 mm in the direction of longest measurement.

The quartz sand should meet the existing standard requirements.

To facilitate grinding of clinker during production of cement, it is allowed to introduce special additives not impairing the quality of cement and in an amount not exceeding 1 % of the mass of cement.

7.3. Specifications for Plugging Cement

In testing plugging cement to determine its physical and chemical properties, use is made of a cement paste prepared with 50 % of water by mass of cement.

The bending strength of beam test pieces measuring 40 × 40 × 160 mm made of the cement paste in a standard manner must, upon hardening for two days, accord with that indicated in Table 22.

Table 22

Requirements for Strength of Test Pieces

Type of cement	Hardening temperature, °C	Bending strength, 10^{-4} MPa, not less than
For "cold" wells	22±2	27
For "hot" wells	75±3	62

The mobility (fluidity) of the standard plugging portland cement paste must be such as to ensure that it will spread over a circle no less than 180 mm in diameter in the standard fluidity cone test.

The setting times of plugging cements should accord with those indicated in Table 23.

Table 23

Required Setting Time of Mixtures

Type of cement	Initial set after mixing	Final set after mixing
For "cold" wells	No sooner than in 2 hours	Not later than in 10 hours
For "hot" wells	No sooner than in 1 hour 45 minutes	Not later than in 4 hours 30 minutes

Plugging cement specimens made of paste of normal thickness must show a uniform change in volume when boiled in water.

The content of sulfuric anhydride (SO_3) in cement must not exceed 3.5 %.

The starting clinker must not contain more than 5 % of magnesium oxide (MgO).

The fineness of ground cement must allow 85 % by mass of the sample to pass through a No. 008 sieve (0.08 mm in mesh size).

7.4. Acceptance Rules

The maximum size of a cement lot (consignment) is set at 200 t. Delivery of cement in a consignment of less than 200 t must not be considered a lot.

Note. If shipped by water, the standard lot of cement may be increased to 300 t.

The manufacturing plant must provide each lot of the consigned cement with a certificate indicating the name of the manufacturing plant, the number of the certificate and of the consignment (lot), the year, month, and date of shipment, the name and address of the consignee, the mass of the lot, the numerical designations of wagons (of ship) and of invoices, type designation of cement, the kind of the additive and its quantity in per cent (according to mean daily production figures), compliance of cement with the requirements set forth in the standard in force, the date of manufacture of samples and the results of their testing, and the numerical designation of the standard in force.

The manufacturing plant, when making the shipment of cement, and the consumer, if necessary, check up the compliance of the cement with the requirements of the standard by employing the following sampling procedure.

To test cement, a sample batch no less than 10 kg in mass is taken from each lot, one sack of cement being taken from every 400 sacks in the lot and a sample of 1 kg weighed out from this sack.

The samples taken from the lot of cement are thoroughly mixed together, quartered and then divided into two equal parts. One of these is subjected to tests as required by the standard, while the other part is numbered and stored in a dry room and in a dry tightly closed container for one month, in case a need for repeated testing should arise.

Note. When the cement is shipped in bulk in trucks the sampling procedure is established by mutual agreement between the supplier and the consumer.

Plugging portland cement, if not in compliance with but one of the requirements stated in the standard, is to be rejected.

Such cement may, by consent of the consumer, be utilized as ordinary portland cement.

7.5. Test Methods

In testing each sample of cement, subject to determination are: its sounding, fineness, flow characteristics of the cement paste, setting time, bending strength.

Physical and mechanical testing of cement is done according to the pertinent USSR standard, with the following complementary procedures and changes.

(a) The temperature in the room where tests are performed, as well as the temperature of water, must equal $22 \pm 2\,^\circ\mathrm{C}$.

(b) By mutual agreement with the consumer, use may be made of sea water for mixing cement, without introducing any changes in the characteristics set up in the standard.

Determination of Normal Thickness (Consistency) and Setting Time of Cement Paste

EQUIPMENT

To determine the normal thickness and setting time of the cement paste in use are the following mechanized laboratory apparatus: a mixer for preparation of the cement paste; a device for automatic recording of the setting time. These devices should satisfy the requirements set forth in relevant specifications.

When the above determinations are made manually, the apparatus employed for this purpose are: a Vicat apparatus (cement needle) with a ring, a bowl, and a small trowel for preparation of cement paste.

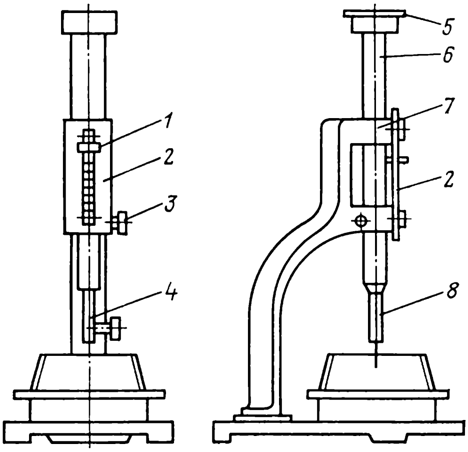

Fig. 62. Vicat apparatus

The mechanical mixer for the preparation of cement paste and the device for automatic recording of the setting time of cement paste are manufactured according to special specifications.

The Vicat apparatus (Fig. 62) has cylindrical metal rod *6* freely moving in the holder of stand *7*. The rod is fixed at the required height with clamping screw *3*, or with some other arrester. The rod carries indicator *1* to read off its displacement over scale *2* fastened to the stand. The value of a scale division is 1 mm.

In determining the normal thickness (consistency) of the paste, cylindrical metal pestle *4* is inserted into the lower part of the rod. When defining the setting time, needle *8* is substituted for the pestle.

The pestle is made of stainless steel with polished surface and the needle, of a rigid stainless steel wire with polished surface and no deflections whatsoever. The surface of the pestle and the needle should be clean. When handling the apparatus, the mass of the moving part of the device, following the replacement of the pestle with the needle, is kept the same by adding complementary weight *5* which is superimposed upon the rod. While using the apparatus shown in Fig. 63 the mass of its moving part is made to remain constant by interchanging the pestle and the needle.

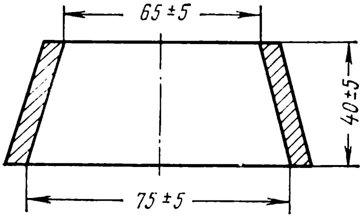

Fig. 63. Ring for Vicat apparatus

The dimensions of the pestle and the needle and their manufacturing tolerances must comply with the figures listed in Table 24.

The recommended masses of basic parts in the Vicat apparatus are given in Table 25.

Table 24

Dimensions of Parts in the Vicat Apparatus, mm

Parts	Diameter	Length
Pestle	10.0±0.02	~50.0
Needle	1.1±0.04	~50.0
Fixed portion of the pestle and needle	5.0	14.0

Table 25

Recommended Mass of Basic Parts in Vicat Apparatus

Parts	Mass of parts, g	Manufacturing tolerances, g
Rod with head and accessories	265	±1.0
Pestle:		
(a) for apparatus with complementary weight	35	±1.0
(b) for apparatus with interchangeable pestle and needle	27.5	±0.5
Needle	7.5	±0.5
Complementary weight	27.5	±0.5

Note. The total mass of the movable part of the apparatus must be within 300±2 g.

The *ring* for the Vicat apparatus and the *plate* upon which the ring is placed during tests should be made of stainless steel, some plastic material, or glass. The shape and dimensions of the ring are indicated in Fig. 63.

When no mechanical mixer is available at the laboratory, a spherically shaped bowl is used for the preparation of cement paste. It is recommended that the bowl should be made of stainless steel and have dimensions indicated in Fig. 64.

It is recommended that the trowel for mixing cement paste should be made of stainless steel. Its main dimensions are shown in Fig. 65.

Determining the Consistency of Cement Paste

By *cement paste* is meant a mixture of cement with water. As normal is considered the thickness of cement paste with which the pestle of the Vicat apparatus immersed into the ring filled with the paste stands 5 to 7 mm above the plate carrying the ring.

The consistency of the paste is characterized by the amount of mixing water expressed in percent of the mass of cement.

Before starting the tests, one should check the free descent of the rod in the Vicat apparatus and also verify the zero reading of the apparatus by bringing the pestle in contact with the plate carrying the ring. Should there be any deviations from the zero reading the scale of the apparatus is to be moved as necessary.

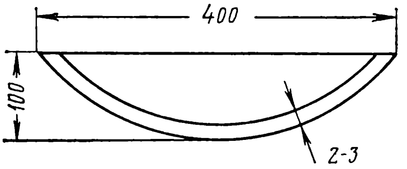

Fig. 64. Bowl for mixing cement slurries

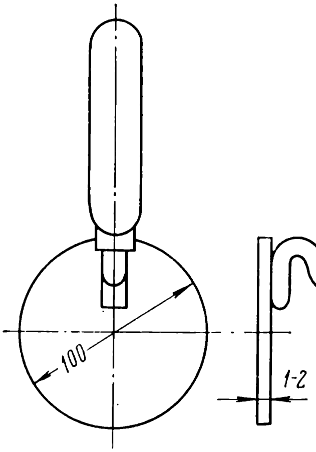

Fig. 65. Trowel for mixing cement slurries

The ring and the plate are greased before testing by applying a thin layer of machine oil.

To prepare cement paste, weigh out 400 g of cement, pour it into a bow (wiped preliminarily with a moist fabric), make a hole in the cement and pour into it water at a go in an amount needed (approximately) to obtain cement paste of normal consistency. After pouring water, fill back the hole with cement and in 30 seconds' time start first stirring carefully and then continue to vigorously triturate the paste with the trowel. The stirring and trituration of cement with water lasts 5 minutes after pouring water.

Note. Where a mechanical mixer is used to prepare cement paste, one should follow the operating instructions supplied with the mixer.

Right after completion of stirring, fill the ring with the paste at a go and shake it 5-6 times by lightly striking the plate against the table. Thereafter, level out the surface of the paste flush with the rim's edges, cutting off the excess paste with a knife wiped with a moist fabric. Immediately thereafter make the pestle of the apparatus contact the surface of the paste in the centre of the ring and fix the rod in place with a clamping screw, following which, by quickly turning off the clamping screw, release the rod and allow the pestle to plunge freely into the paste. After a lapse of 30 seconds from the instant of the rod's release, read out the immersion on the scale. During measurement, the ring with the paste should not be subjected to any impacts.

Should the consistency of cement paste prove inadequate, change the amount of water and mix the paste anew by trying to obtain the immersion of the pestle to a depth indicated in the standard. The amount of water added to obtain a paste of normal thickness, expressed in percent of the mass of cement, is determined to an accuracy of up to 0.025 %.

Determining the Initial and Final Set of Cement Paste

Before testing, it is necessary to check the free descent of the rod in the Vicat apparatus and also verify its zero reading. In addition to this, check on the cleanliness and the absence of deflections (bucklings) of the needle. Make the needle contact the surface of the cement paste put into the ring, and fix the rod in this position with a clamping screw, then release the rod, following which the needle should freely plunge into the paste. At the start of the test, while the paste is still liquid, the needle may be held back gently while sinking into the paste, to prevent its striking heavily against the plate. As soon as the paste gets so thick as to exclude the danger of the needle becoming damaged — allow the latter to go down freely. The instant of the initial set is to be determined with the free descent of the needle.

The needle is plunged into the paste every 5 minutes prior to the initial set and every 15 minutes later on. The ring is moved aside a little each time after immersion, to prevent the needle to plunge at one and the same point. After each immersion the needle is to be wiped dry.

At the time of testing, the ring should be kept in a shade, away from draughts, and not subjected to rocking.

The initial set of cement paste is considered to be the time lapsed from the start of mixing (pouring in of water) till the moment when the needle stands 1 to 2 mm above the plate. *The final set of cement paste* is characterized by the time passed from the beginning of mixing till the moment when the needle sinks into the paste to a depth of not more than 1 mm.

Note. Where the initial and final set of cement paste are determined with the aid of an automatic setting time recorder, one should follow the operating instructions supplied with the instrument.

Sounding Tests of Cements

APPARATUS

It is recommended that the *small tank* for tests by boiling should be used in conjunction with a water level controller (Fig. 66). This purpose is served by a vessel filled with water and provided with movable vertical tube that allows the water level in the tank to be set at the required height. The tank houses a removable grilled rack to carry cement cakes, and the distance between this rack and the bottom of the tank should be not less than 2 cm. The water level in the tank is to stand 4 to 6 cm above the cakes.

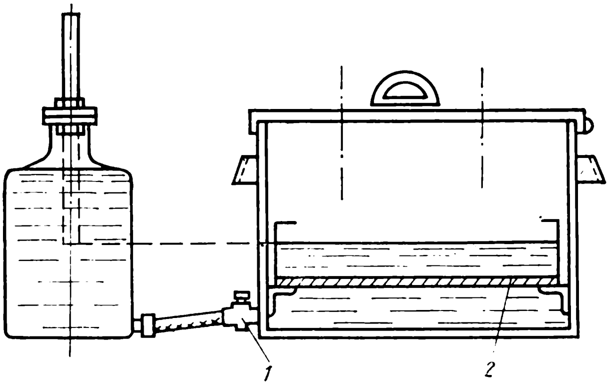

Fig. 66. Boiling test tank
1 — cock; 2 — grill

The water in the tank is brought to the boiling point over a period of 30 to 45 minutes on any heater.

The *autoclave* for sounding tests of cement containing over 4.5 % of MgO is to be designed and tried out at 2 MPa by a pertinent engineering body; it should be provided with a pressure regulator and a safety valve. The electric heater power should secure the rise of pressure in the autoclave under total load to 2 MPa over a period of 1.5 to 2 hours. After cutting off the electric heater, a pressure of 2 ± 0.05 MPa must remain for 1 hour. The autoclave is to be fitted out with a valve for discharge of air and relief of pressure remaining at the end of the 1st hour of cooling, and with a 3 MPa pressure-gauge having a reading accuracy of up to 1 % and a valve of a scale division not exceeding 0.1 MPa.

The autoclave should have a special rack to accommodate specimens above the water level.

Baths with a hydraulic seal are better to be made of galvanized steel sheets. The baths have gratings placed inside them to carry specimens. The gratings should always have water underneath them.

PREPARATION OF SPECIMENS

To make sounding tests of cement, a paste of normal consistency is prepared.

For this purpose, weigh out 4 lots of the paste weighing 75 g each and place each lot in the shape of a ball on a glass plate, preliminarily smeared with machine oil. Then shake the plates for as long as the balls spread out and assume the shape of cakes measuring 7 to 8 cm in diameter with a thickness of about 1 cm in their middle. To obtain sharp edges and evenly rounded surface, smooth the cakes over, starting from the outer edges and proceeding towards the centre, with a water-wetted knife. The four cakes prepared in this way are kept for 24 ± 2 hours (from the moment of their preparation) in the bath with a hydraulic seal and then tested.

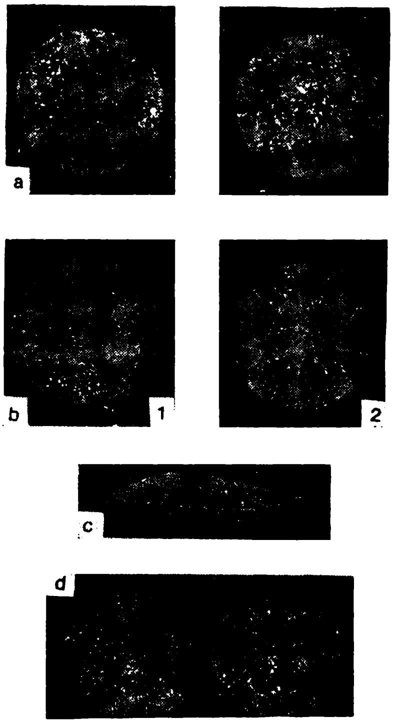

Fig. 67. Cement cakes after sounding test

(*a*) sound specimens; (*b*) failed specimens; *1* — breakage; *2* — radial cracks; (*c*) failed specimen (warpage); (*d*) sound specimens (shrinkage cracks)

Test Procedures

Tests by boiling. After expiration of 24 ± 2 hours after mixing, take two cement cakes off the plate, withdraw them from the bath and place on the grating in the small tank filled with water (see Fig. 66). Thereafter bring the water in the tank to the boiling point and keep it simmering for 4 hours, following which cool the cakes in the tank and inspect them visually immediately after their removal from water.

Autoclaving. Place two cakes that have stood the preliminary test by boiling and are intended for autoclaving onto the grating of the autoclave, 12 to 15 % of the latter's volume being preliminarily filled with water which, however, should not cover the cakes. Then close the cover of the autoclave by turning on alternately the contralateral nuts. The electric heater of the autoclave is to be switched on with the air-discharge valve open. When a steam jet starts issuing from the valve, it should be closed. The pressure in the autoclave should be brought to 0.05 MPa over a period of $1^3/_4$ to 2 h. Thereupon, the electric heater of the autoclave is to be cut off and, in the course of 1 hour, the autoclave pressure should be brought to the normal value by using the valve. After cooling the autoclave, its cover is opened and the cakes are inspected.

The cement is considered to comply with the sounding requirements if the front side of the tested cakes does not present any radial cracks reaching the edges, or a system of minute fissures visible by the naked eye or with a magnifying glass, as well as some deflections (bucklings) or an increase in the volume of the cakes. The presence of bucklings or warpings is ascertained with the help of a rule applied to the flat surface of the cake. Specimens of cakes which did or did not stand the sounding test are shown in Fig. 67.

Notes: 1. Cracks due to drying-up that become apparent at times during the first 24 hours after preparation of the cakes but fail to reach their edges do not signify the poor quality of the cement, provided their back face does not exhibit any radial cracks reaching the edges and on knocking them one against the other the cakes produce a ringing sound.

2. Radial cracks appearing sometimes on the cakes that have stood the boiling test and have been left for some time in the open air come as a result of internal shrinkage stresses and are not to be regarded as a poor-quality criterion.

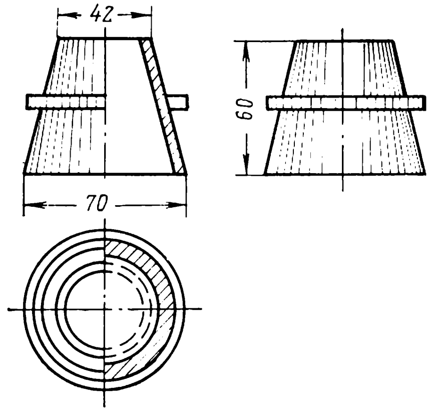

Fig. 68. Standard fluidity test cone

Determination of Fluidity (Spreadability) of Cement Paste

The fluidity, or spreadability, of cement paste is determined with the aid of an apparatus in the form of a truncated cone (Fig. 68) 120 cm^3 in volume, 36 ± 0.5 mm in top clear opening diameter, 64 ± 0.5 mm in base clear opening diameter, and 60 ± 0.5 mm in height. The mass of the cone must be at least 300 g. The cone is made of a stainless metal and its inside surface is polished.

When testing cement paste for fluidity, the cone is placed on a piece of glass arranged horizontally, a sheet of paper with a series of concentric circles traced on it at 5-mm intervals being placed under the glass. The diameter of the largest circle in the series is 250 mm and that of the smallest one, 100 mm. Drawn on the paper is also another circle whose diameter is equal to the outside base diameter of the cone, i. e., 70 mm, which enables the cone to be placed exactly in the center.

To test cement paste for compliance with the standard fluidity requirements, weigh out 500 g of cement, transfer it into a spherical bowl, and add 250 g of water all at once, the amount of water being weighed out or measured off to an accuracy of 0.5 g or 0.5 ml.

Vigorously stir up the mass in the bowl for 3 min, fill the cone to the brim with the paste thus obtained, and then lift the cone vertically.

To determine the fluidity of the paste, read off its maximum and minimum spread diameters and then take the arithmetic mean of these two readings.

Prior to the test, wipe the cone clean and dry.

Determining the Setting Time of Cement Paste for "Cold" Wells

Immediately after completing the fluidity test, vigorously agitate the remaining cement paste for 0.5 min and pour it in the Vicat ring. Level out the surface of the paste with a knife one hour after mixing and leave it in the open air at 22 ± 2 °C.

The Vicat needle is immersed in the paste for the first time one hour after mixing, the subsequent immersions following at a rate of not less than every 15 minutes.

Determining the Setting Time of Cement Paste for "Hot" Wells

To determine the setting time of the paste, pour the latter in the Vicat ring, level out the paste surface with a knife and cover the paste surface with a glass plate. Immediately thereafter immerse the ring with the paste into a water thermostat filled with fresh water maintained at a temperature of 75 ± 3 °C. This purpose can be served by water thermostats uniformly heated by any source of heat which should not contact the ring. The ring together with the cement paste is withdrawn from the thermostat, tested with the Vicat needle and then dipped anew into the thermostat.

The first immersion of the needle in the paste is done one hour after mixing and the subsequent ones, every 5 minutes till the end of the test.

The initial set of the cement paste is counted by the time passed since the start of mixing (the moment of adding water) till the instant when the needle stands 1-2 mm away from the plate. By the final set of the cement paste is meant the time lapsed from the commencement of mixing till the moment when the needle dips into the paste down to a depth of not more than 1 mm. When determining the setting time, the ring should not be subjected to impacts.

Determining the Bending and Compressive Strengths of Standard Beam Test Pieces Made of Cement Slurries

APPARATUS

Collapsible moulds of steel or cast-iron for preparation of specimens are shown in Fig. 69. The individual components of the moulds should be numbered so as to facilitate their assembly.

The Brinell (ball) hardness number of the mould metal should be at least 140. The mould dimensions (in mm) are as follows:

Width B	40
Height H	40
Length L	160
Manufacturing tolerances	± 0.02

The longitudinal and transverse walls of the moulds should be ground on both the top and bottom end faces and should closely fit the bottom plate. The walls should be tightly pressed against the bottom plate by clamping bolts, and the angle between the walls and the bottom plate should be 90 ± 0.5°.

The mould dimensions should be checked at least once every six months and the results entered in a log-book. If the mould dimensions deviate more than ±0.2 mm from the standard values, the moulds should be discarded.

Appliances for cleaning the moulds should be made from such materials (e. g., copper, wood) as are softer than the metal of the moulds. The mould extension piece used for making standard beam test pieces should tightly fit the top edges of the mould walls.

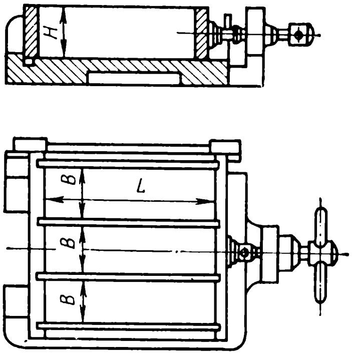

Fig. 69. Collapsible mould for preparing standard beam test pieces

Bending machine for testing standard beam test pieces. To test standard beam test pieces for bending strength, use may be made of any suitable testing machine, provided that it meets the following requirements. The breaking load reading accuracy of the machine should be no worse than 5 N/s. The bearing and pressure rollers should be strictly parallel to one another. The pressure roller should lie in the midplane between the bearing rollers. The distance between the centers of the bearing rollers should be

100 mm and the rollers themselves should be 10 mm in diameter. The pressure roller holder should be equipped with a ball-and-socket bearing. All the rollers should be made of a hard stainless steel.

Standard beam test pieces may also be tested in the Michaelis tensile strength testing machine (Fig. 70), but in this case, the holders for mortar briquettes have to be replaced by a flexure testing attachment complying with the above requirements. The loading bucket must be no more than 600 g in mass and the test load must be provided by shot 2 to 3 mm in diameter falling in the bucket at a uniform rate of 100 g/s.

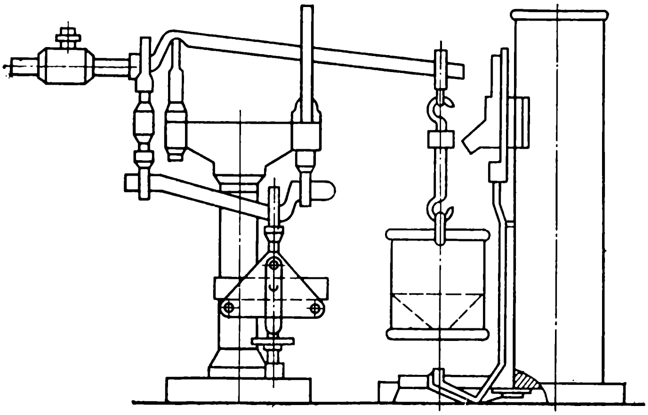

Fig. 70. Michaelis testing machine

It is recommended that the compression machine for testing the halves of the standard beam test pieces that have undergone bending tests should have a maximum load capacity of 500 kN.

Note. Considering the small size of the standard beam test-piece halves subject to compression tests, it is recommended that the compression machine should be equipped with bearing plates no more than 10 × 10 cm^2 in size.

The plates should be provided with a centering arrangement to locate the pressure plate serving to transmit the test load to the specimen.

The plates for transmitting the test load to the beam test-piece halves (see Fig. 71) should be made of stainless steel with a hardness of 60 Rc and should have a flat ground surface 40 × 62.5 mm^2 in size. Freshly manufactured plates should be flat to within 0.02 mm and plates that have already been in use, to within 0.05 mm.

The mould for the preparation of the test pieces, assembled and complete with the top extension piece, is placed on a marble, glass, or metal plate, and the inner surfaces of the mould and of the bottom plate are smeared with machine oil. To prevent water escaping from underneath the mould, the lower portion of its walls is coated on the outside with solid oil or some other grease.

The cement paste used for the preparation of the test pieces is mixed by hand in an amount sufficient to make three specimens.

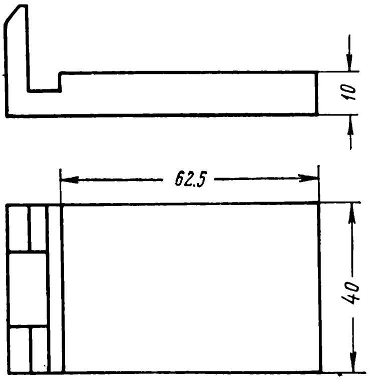

Fig. 71. Bearing plates for compression tests of standard beam test-piece halves

To accomplish this, 1600 g of cement and 800 g of fresh water are mixed in a spherical bowl for 3 minutes.

While being continually stirred, the paste is poured in the moulds. The recesses are filled consecutively in two steps: first, the paste is poured in to fill the mould roughly to one half of its height and at the second go the recess is filled with the paste flush with the brim of the top extension piece without overflow. The paste should be poured in as quickly as possible. One hour after mixing the top extension piece is taken off and the surface of the cement paste is smoothed out with a knife.

Storage of Standard Beam Test Pieces Intended for Testing at 22 ± 2 °C

During the first 24 hours following their preparation, the test pieces in the moulds are kept in a closed cabinet or in a bath provided with a hydraulic seal (at a relative humidity of 80 to 90 %). In 24 ± 2 hours after mixing, the test pieces are cautiously freed from the moulds. Thereupon, the test pieces are numbered and placed straight away into a basin or a bath with a hydraulic seal for storage until testing. The water in the basin is to be changed every 14 days.

The test pieces should be kept in a single row at a distance of not less than 1 cm from one another and the water level should stand no less than 2 cm above the surface of the test pieces.

Note. In cases where a test piece, upon its removal from the mould, is found to have shrunk more than 1 mm on cross section, the test piece should be marked in a suitable manner and its cross-sectional area should be measured prior to testing.

Storage of Standard Beam Test Pieces Intended for Testing at 75 ± 3 °C

Test pieces prepared as indicated above are covered with a glass plate, after removal of the excess paste with a knife, and put into a thermostat filled with fresh water having a temperature of 75 ± 3 °C. On expiration of 24 ± 2 hours after mixing, the test pieces are removed from the thermostat, quickly freed from the moulds, numbered and immediately placed into the same thermostat for further storage.

The water in the thermostat is changed every 6 days, it being preliminarily heated up to 75 ± 3 °C. The amount of water evaporated during storage of the test pieces is compensated for by adding new portions of water preliminarily heated up to 75 ± 3 °C.

Test Procedures

Bending Strength Tests

The test pieces are installed on the bearing rollers of the bending arrangement in such a manner as to make their horizontal faces (when in the mould) stand upright.

Where the Michaelis testing machine is used, one should check, prior to the test, on the correct position of the knife-edge supports of the machine and its balance with the loading bucket dismounted. In this position, the top face of the upper beam must register with the reference line or arrow marked on the yoke.

After putting the test piece in place, the upper beam is raised with the aid of the adjusting screw to such a height as would make it come as close as possible to the equilibrium position at the moment the test piece fails.

Once the test piece is in position, the load bucket is gradually charged with shot from the bin of the machine. As soon as the mass of the bucket charged with shot reaches the breaking load value, the test piece fails and the bucket falls on the bin gate pedal, thus stopping the shot feed into the bucket. The shot feed rate must be 100 ±10 g per second. The breaking load is found by weighing the shot-charged bucket accurate to within 10 g, and the bending strength (in MPa) of the test piece is then found by the formula

$$\sigma_{bend} = (3Pl/2bh^2)\,k \tag{40}$$

where P is the mass of the shot-charged bucket, kg; l is the distance (span) between the bearing rollers, cm; b is test-piece width, cm; h is the test-piece height, cm; k is the coefficient determined by the leverage of the machine.

For a beam test piece measuring 4 × 4 × 16 cm, a span of 10 cm, and a machine leverage of 1 : 50, the above formula takes the form

$$\sigma_{bend} = 11.7P$$

The bending strength of cement stone is determined as the arithmetic mean of the larger two of three test results.

Compressive Strength Tests

The six beam test-piece halves resulting from the bending tests are subjected to compression tests.

Each test-piece half is placed between two bearing plates so that its vertical side faces (when in the mould) are in contact with the plates and its flat end face tightly fits the rests on the plates

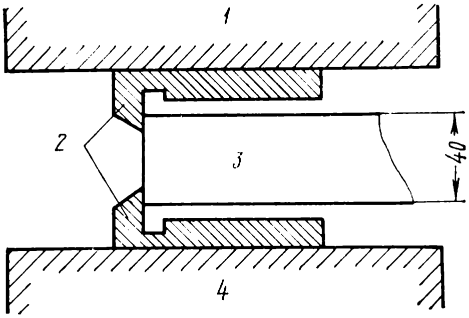

Fig. 72. Illustrating the compression tests of standard beam test-piece halves
1 — compression testing machine pressure plate; 2 — bearing (compression) plates; 3 — standard beam test-piece half; 4 — compression testing machine bed

(Fig. 72). The bearing plates with the specimen clamped between them are then compressed in a compression testing machine, the load on the specimen being increased at a rate of 2 ± 0.5 MPa/s.

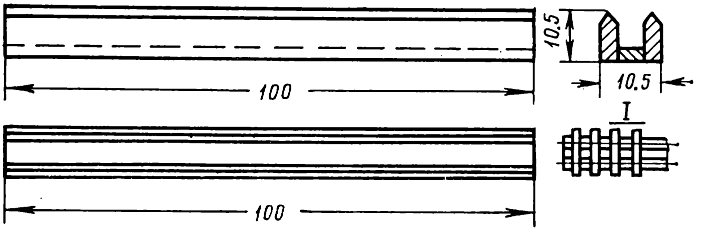

Fig. 73. Wooden stand for cooling cement specimens
1 — arrangement of standard beam test pieces on the stand

The compressive strength of each specimen is found by dividing the breaking load (in N) by the effective area of the bearing plates (in cm^2), i. e., by 20 cm^2.

The compressive strength of cement stone is calculated as the arithmetic mean of the larger four of the six test results.

Test pieces that have been kept in water at 22 ± 2 °C should be removed from water 48 ± 2 h after mixing and tested within the next 15 min. Immediately before the test, the specimens must be wiped dry.

Test pieces that have been held in a thermostat at 75 ± 3 °C should be removed from the thermostat 48 ± 2 h after mixing, cooled for 2 h 30 min at room temperature, and then tested.

The test pieces are cooled on special wooden stands (Fig. 73).

The chemical analyses of cement (for SiO_2 and MgO) are performed in conformity with the USSR Standard GOST 5382-73.

7.6. Transportation and Storage

Cement is to be shipped in paper sacks or in special cement-carrying trucks.

For sacking, use should be made of multilayer (four-, five-, or six-layer) paper sacks meeting the requirements of the USSR Standard GOST 2226-75.

Note. By mutual consent of the parties the cement may be transported in containers of different types.

The paper sacks should carry the following designations: the name of the manufacturing plant, name of the cement, numerical designation of the consignment, year, month, and day of sacking.

The cement shipped in cement-carrying trucks should be forwarded with a document containing the above information.

During its transport and storage, cement should be protected against moisture and contamination with impurities.

7.7. Determining the Permeability of Cement Stone

The permeability of cement stone is commonly determined by means of the procedures and apparatus (ГК-5 Model setup) intended for determining the gas permeability of core samples retrieved from drill holes. The specimens that have to undergo testing should be kept under conditions matching the effects of temperature and pressure for which the composition (formulation) of the plugging mixture is chosen at the laboratory. For all practical purposes, it is expedient that the specimens for establishing the permeability be turned from the halves of the standard beam test pieces that hardened in baths or autoclaves under the required conditions and over specified time-periods and were intended for tests in bending.

The ГК-5 Model setup (Fig. 74) consists of the following main units: the ЗС-8 Model screw clamp *17* to hold the test piece, the ХК-4 Grade PVC tube to dry the test gas, the МЖ-10 Model liquid-column (U-tube) manometer *10* to measure the gas pressure upstream of the test piece, the СР-1 Model measuring (graduated) flask *12* to measure the rate of gas flow through the test piece and the gas pressure downstream of the test piece, pressure regulators *2* and *16*, and an air (gas) cylinder with valve *1*.

The test for the permeability of cement stone starts with the preparation of the test piece (specimen). The test piece (beam or cube), hardened under specified conditions, is machined to obtain a cylinder with square end faces. Weak specimens require special treatment which consists in giving the specimens a coat of litharge or Wood's alloy. Once the test piece is finished, the test setup is checked for leaks and the specimen is tested. The procedure is as follows.

1. Close low-pressure requlator valve *3* by turning it counterclockwise and then open valve *1* of the gas cylinder to admit the test gas to the high-pressure requlator controlled by valve *2*, the gas pressure admitted to the regulator being registered by pressure gauge *16*. Keeping low-pressure regulator valve *3* closed, open high-pressure regulator valve *2* by turning it clockwise until low-pressure gauge *4* reads 0.1 to 0.12 MPa.

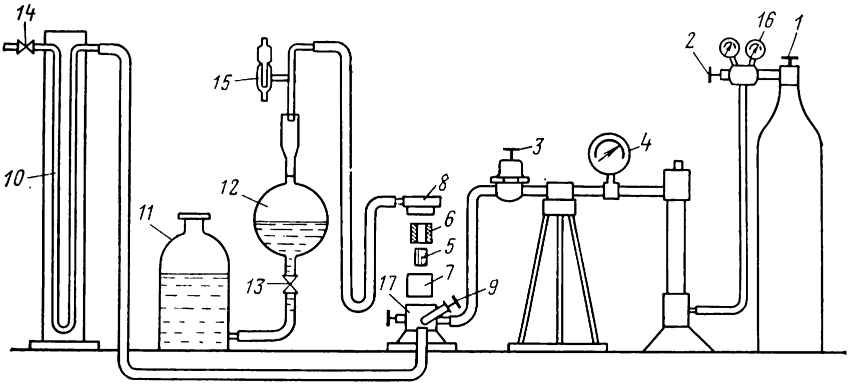

Fig. 74. ГК-5 Model setup for determining permeability of core samples and cement specimens

2. Determine the dimensions of the sample, dried beforehand until its mass remains constant (except for special cases), accurate to within 0.1 mm and enter the average diameter d_{av} and average length l_{av} of the specimen in a log-book. Insert specimen *5* into rubber casing-sleeve *6*, place the casing with the specimen into steel cylinder *7* with a screen at the bottom, install the cylinder on clamp *17*, cover it with clamp head *8*, and fasten with screw *9*.

3. With valve *14* kept open, pour the coloured water contained in vessel *11* into measuring flask *12* until its level in the flask stays above the top graduation mark.

4. Gradually open low-pressure regulator valve *3* by turning it clockwise until U-tube manometer *10* reads a pressure differential of 0.4 MPa. Close valve *14* and simultaneously open valve *13* to let the test gas (nitrogen or air) pass through the test piece. This causes the water level in the measuring flask to drop. The moment the water meniscus in the flask comes to register with

the top graduation mark, start the hand of a stop-watch and follow the water level in the tube of indicating device *15*, which must coincide with the overall water level in the device, the match being achieved by manipulating valve *13*. Any recurrent pressure surges registered by U-tube manometer *10* point to a faulty pressure regulator and should this be the case, the test should be discontinued. As soon as the water meniscus goes past the bottom graduation mark of the measuring flask, stop the hand of the stop-watch, open valve *14*, and close valve *13*. Read the stop-watch to determine the time it has taken the water meniscus in the measuring flask to descend from the top to the bottom graduation mark and enter this time in the log-book.

5. In the course of the test, read a barometer and a thermometer to determine the ambient pressure and temperature and enter these readings in the log-book. Using the ambient pressure and temperature values, find L with reference to the standard tables. Then calculate d^2_{av} and l_{av}/d^2_{av} and find the permeability of the test piece (in μ^2) by dividing the magnitude of $L(l_{av}/d^2_{av})$ by the time it has taken the water meniscus in the measuring flask to descend from the top to the bottom graduation mark.

6. Close low-pressure regulator valve *3*, remove the test piece from the screw clamp, close gas cylinder valve *1*, and open high-pressure regulator valve *2* to release the test gas from the system.

Repeat the test at a higher pressure.

Example

Test piece code	T, °C	p, MPa	l_{av}, cm	d_{av}, cm	d^2_{av}, cm^2	l_{av}/d^2_{av}	p_{max}, MPa	L, cm	t, s	$L(l_{av}/d^2_{av})$	k, $10^{-3}\mu^2$
	28	76.1	2.97	2.91	8.47	0.35	0.4	32110.7	9000	11238.7	1.2

Laboratory Work No. 4

In accordance with the USSR standard for plugging portland cement, determine:

(1) fluidity of cement slurry;

(2) setting time of cement slurry at 22 ± 2 °C and 75 ± 3 °C;

(3) bending and compressive strengths of cement stone at 22 ± 2 °C and 75 ± 3 °C;

(4) permeability of cement stone.

Chapter 8

Adjusting the Properties of Cement Slurry and Cement Stone

A cement slurry intended for plugging oil wells should possess a number of properties that mixtures of plugging portland cement and water alone lack.

Slurries intended for cementing jobs in actual wells must be tried out in situations matching the conditions existing in the wells. Should the properties of the slurries and stone mismatch actual conditions, these properties have to be changed and the composition formulation of the slurries corrected.

These requirements must be always complied with, especially when cementing deep high-temperature wells.

To shorten the setting or thickening time of slurries prepared of plugging cement, application is made of accelerating agents and, conversely, to prolong this time, retarders are used. Among cement setting accelerators, it is calcium chloride, $CaCl_2$ (2 to 3 % of the mass of cement) that is employed in most cases; the same purpose is served by aluminium chloride, $AlCl_3$ (up to 10 %) and by small dosages of sodium chloride, NaCl (2 to 3 %). When added in large amounts, NaCl tends to slow down the structurization in cement slurries. The same reagents are put to use in speeding up the setting of cement mixtures in plugging wells driven in permafrost areas.

The said accelerating agents (as well as retarders) are preliminarily dissolved in water and mixed with cement. The resulting cement mixture is pumped into the well.

In principle these agents may be added to the cement in the course of grinding or they may be intermixed with aggregates. This method, however, is a complicated one, for its implementation requires special conditions and there is no guarantee of obtaining a mixture displaying uniform properties from the entire lot of cement. For this reason it is the first method that is usually resorted to. Considering that the method is realized under field conditions it is indispensable that the properties of the composition of the cement mixtures obtained in the laboratory and in the field be exactly the same.

8.1. Cement Setting Retarders

Because of rising bottom-hole temperatures and pressures and accelerated rates of cement setting, a search for and selection of adequate retarding agents become a matter of necessity. Although several scores of retarders are known today (boracic acid, starch, ferric sulphate, sugar, syntan, etc.) only few of them find practical use. These include: calcium lignosulfonates, spent sulfite-yeast liquor (SUL), carboxymethylhydroxyethyl cellulose (CMHEC), modified lignine retarder (MLR), carboxymethylcellulose (CMC), spent sulphite-alcohol liquor (SAL), commercial cream of tartar, tartaric acid (TA), hypan, wood-chemical polyphenols, and others. Furthermore, application find oxidized cellulose, sodium tannate, dextrin (starch gum), and pyrogallic acid.

SAL is distinguished by inconsistency of its retarding properties and does not produce an equal effect on all types of cement. It has a greater effect on the brands of cement turned out by the Karadag plant, and is less active with the cement grades produced by the Novorossiysk plant. SAL should meet technical requirements for its production, acceptance, and storage; SAL having a concentration of 50 % has a density of 1.26 to 1.28 g/cm^3.

Table 26 gives the relation between the density of SAL and the concentration of its solid phase.

Table 26

Density of SAL as a Function of Its Solid Phase Concentration

Density, g/cm^3	Solid phase concentration, %	Density, g/cm^3	Solid phase concentration, %	Density, g/cm^3	Solid phase concentration, %
1.01	2	1.12	23.6	1.25	45
1.02	4	1.13	25.4	1.26	46
1.025	5	1.14	27.4	1.27	48.1
1.03	6	1.15	29.2	1.28	49.7
1.035	7	1.16	31	1.29	51.3
1.04	8	1.17	32.6	1.3	52.6
1.05	10	1.18	34	1.31	54.3
1.06	12	1.19	35.6	1.32	56
1.07	14	1.2	37.1	1.33	57.4
1.08	16	1.21	38.6	1.34	59.1
1.09	18	1.22	40.2	1.35	60.6
1.1	20	1.23	42	1.36	61.3
1.11	21.6	1.24	43.4		

The solid phase concentration of SAL also influences its effectiveness in retarding the setting of cement slurries.

Slurries prepared of stale cement lend themselves more readily to the treatment with spent sulphite-alcohol liquor than those made of a freshly ground cement.

It is recommended that not more than 1.5 per cent of SAL by mass of cement be introduced into the mixture, but in most cases foaming starts already with 0.5 % of SAL.

The period of action and the stability of the adsorption layers, as well as the overall retardation effect of surfactants are to be determined directly on cement slurries by finding their setting time, viscosity, and other properties.

In all cases of investigations and industrial usage, the retarder is dissolved in water and this is then used for mixing with cement. The effect exerted on cement by retarders depends on many factors, *viz.*, the time and conditions of cement storage, its chemical and mineralogical composition, properties of the retarder itself, etc. Therefore, any cement slurry formulation should be compiled shortly before the actual operation and only with the water and the brands of cement and retarding agent that will be made use of in cementing. It is common that, while once selected a slurry composition yields a suitable initial set, with the cement kept in storage for a certain time the setting time of the slurry becomes, as a rule, longer.

With greater SAL dosages, the setting time of cement slurries decreases. Rising temperature and pressure require an increased consumption of SAL, for then the adsorption processes proceed at a quicker pace, with hydrolysis and hydration of cement going on faster.

SAL thins out cement slurries and for this reason it is widely used for bringing down the water-cement ratio and increasing the density and even for accelerating the setting rate of the slurries (at low water-cement ratios). At the same time, SAL makes cement slurries foam intensively, thus obstructing the proper execution of cementing jobs. At times there builds up so much froth in the receiving tank of cementing units that it becomes impossible to continue the operation, for the pumps start drawing in air and it becomes difficult to estimate the volume of the fluid pumped in. As a froth breaker, it is expedient to apply a neutralized contact black, a small amount of which (0.1 to 0.3 %) reduces the effect of foaming without affecting the setting time. Other reagents may also be employed for this purpose, among which a good froth breaker is a calcium salt of naphthenic acids (0.1 to 0.2 %) developed in this country.

Carboxymethylcellulose (CMC) shows the ability to retard the setting of cement slurries at temperatures up to around 130 °C and high pressures. It is manufactured in several modifications differing in viscosity and stabilization capacity. CMC does not cause foaming of cement slurries; it is fairly stable and very active, and its cement setting retardation effect is proportional to its amount taken for treating the slurry. CMC is put out in the form of a white granulated powder which has no odour or taste. Its density is 1.6 g/cm^3. CMC is used in the form of aqueous solutions 5 to 10 % in concentration, prepared at a temperature of 60 to 80 °C. Agitation (preferably by hydraulic means) speeds up the process of dissolution of CMC.

The preparation of a CMC solution at the drilling derrick (especially in winter months) proves somewhat difficult because of the absence of special baths. Therefore, it is advisable that CMC should be dissolved in water in advance and delivered to the drilling derrick as ready-made solution.

The CMC solution is a stable colloidal solution that does not lose its retarding properties with the passage of time. Already with an addition of 7 to 10 % of CMC, the solution assumes the form of a very tough mass. The reason for such an abrupt rise in the viscosity of the solution is to be sought in the very nature of CMC, which, as it is known, belongs to the category of high-molecular compounds. The molecules of the latter are stretched out into long chains that form in a solution spatial structural lattices whose loops contain entrapped ("immobilized") water. It is but natural for such a system to be greatly thickened, but liable to lose some of its stability during stirring. Therefore, with the dosage of CMC increased beyond 0.3 to 0.5 % the mobility of cement and cement-sand slurries decreases quite materially.

Pertinent tests have shown that in order to obtain cement slurries with suitable setting rates, the operating dosages of CMC should not exceed 1.0 % (by mass of cement). The structural lattices forming in cement slurry when it is treated with CMC become subject to destruction which intensifies with rising speed of stirring or movement of the slurry. The entrapped water gets liberated and the slurry becomes thinner. The molecular chains are oriented along the stream. During stoppages of the process of pumping the slurry, the structural lattice can reappear, depending upon temperature, and for this reason higher pressures must be built up in order to restart the process.

With growing temperature, the viscosity of cement slurries treated with CMC somewhat decreases (here is meant the viscosity due to the CMC treatment), this being explained by the greater intensity of the molecular motion and difficult structurization. The possibility of formation of structural lattices can be reduced by introducing SAL.

At 22 °C, CMC in an amount of 0.3 % so effectively retards the setting of cement slurry that the latter does not harden for as long as two and a half days. At 75 °C, 0.5 % of CMC makes it possible to obtain slurries with their setting time ranging from 7 to 9 hours.

CMC starts to decompose at a temperature of 120 to 130 °C, and so it is unfit for use at temperatures above 140 °C.

CMC tends to somewhat reduce the mechanical strength of cement stone.

CMC retards more effectively the setting of slurries made of stale cements. Slurries prepared of freshly-ground cements sometimes lend themselves with difficulty to the CMC treatment. With some cements, this treatment becomes ineffective already at 110 to 120 °C and even considerable amounts of the agent retard the setting for no longer than around 1 h.

Investigations have demonstrated that at a temperature close to 170 °C and a pressure of about 60 MPa CMC, SAL, starch, and other high-molecular organic retarders are subject to decomposition.

Wood-chemical polyphenols (WCPP) have also been suggested as setting retarders for cement slurries. An analysis into the effect produced by these agents as retarders has demonstrated an irregular inhibitory action, namely, additions of the agents in an amount of up to 0.3 % at 75 °C produce a retarding effect, while in larger proportions (up to 1 %) they retard the final set and accelerate the initial set. At higher temperatures the retarding action of WCPP declines considerably.

The All-Union Oil Research Institute has proposed a combined setting retarder for cement slurries. It is made up of hypan (a copolymer of sodium polyacrylate, polyacrylamide, and polyacryl-nitryl), CMC or SAL with a bichromate (sodium bichromate $Na_2Cr_2O_7$ or potassium bichromate $K_2Cr_2O_7$), whose quantities are chosen to suit the concrete conditions and in conformity with the selected binders and aggregates.

Separate introduction of hypan or any of the bichromates affects but little the setting time of plugging mixtures. But a carefully chosen joint proportioning of these agents results in a substantially increased setting rate of these mixtures and contributes to their thinning. Bichromates raise the retarding action of hypan, CMC and SAL at high temperatures and pressures. The ratio of hypan (CMC or SAL) to bichromate is usually taken at 2 : 1. The most widely employed dosages in treating cement slurries for 110 to 140 °C and pressures of 30 to 50 MPa are 0.4 to 0.6 % for hypan and 0.2 to 0.3 % for the bichromates.

A new setting retarder (Grade Л-6) for cement slurries to be used at temperatures of up to 170 °C has been developed in this country. It is obtained through hydrolysis from pentosanes contained in maize stumps in an amount of 29 % expressed in terms of dry substance and also in waste products of food industry. The Л-6 Grade retarder is a complex mixture of different acids and hydroxyacids (oxalic, trioxyglutaric, xylonic, and others), as well as of their salts. An addition of 1.5 to 2 % of Л-6 (by mass of cement) helps ensure the initial set of the slurry at 170 °C and pressure of up to 70 MPa in 2 to 3 hours and the final set, in 3 to 4 hours. The action exerted by the agent on many properties of cement slurry undergoing hardening and on the stone developing therefrom is analogous to that produced by tartaric acid (TA), although it costs but a fraction of the latter.

Also developed in the USSR is a cement setting retarder produced from fruit-processing wastes (grape, apple, and cherry plum residues). A new process has been worked out for the manufacture of another setting retarder for plugging mixtures, based on wine yeast (WY). The process consists in dissolving sodium hydrate in water, followed by addition of WY. After stirring for 2 to 3 hours, there forms a stable solution without any sediment. The ready-made reagent is capable of plastisizing plugging mixtures and of reducing their water loss.

Laboratory investigations prove that a cement slurry containing 3 % of the retarder has an initial set of 7 hours in tests at 130 °C and a pressure of 60 MPa, with no deterioration in the physical and mechanical properties of the stone.

Tartaric acid (TA) — $C_2H_2(OH)_2 \cdot (COOH)_2$ —is a stable cement setting retarder suitable for use at temperatures of 150 °C and higher and considerable pressures. It retards the setting of cement slurries more effectively than other popular retarders. Tartaric acid (dextrotartaric, or dehydroxysuccinic, acid) is a colourless solid having no odour. Its melting point is 170 °C. With rising temperature its solubility in water increases. At 0 °C, 115 parts of it are dissolved in 100 g of water and at 100 °C, 343 parts. With protracted heating at 180 °C, the acid decomposes. Tartaric acid exists in a free state or in the form of salts in various plants. The main source of tartaric acid are waste products of the wine industry.

An addition of 0.5 % TA (by mass of cement) at 90 °C retards setting so much that slurry remains liquid after expiration of 36 hours. At 150 °C and a pressure of 50 MPa, the same amount of TA retards the initial set by up to 1 hour 30 minutes. To obtain a mixture with an initial set of 1.5 to 2 hours at 170 °C and a pressure of 60 MPa, 1 to 1.3 % of TA are needed. Tartaric acid as a retarding agent is characterized by a low selectivity: a large number of cement samples yield roughly similar results. In the course of setting of TA-treated cement slurries at high temperatures and pressures, a crust forms at the top of the specimen, mainly because of the retarder's precipitation, although the whole of the specimen has not yet hardened. The initial set of a cement slurry with an addition of 1 % tartaric acid at 200 °C and a pressure of 70 MPa takes place, as a rule, not later than in 50 minutes.

The lengthening of the setting time up to 1 hour 30 minutes under the said conditions is secured by the use of a combined agent (TABA) consisting of 1.25 % of TA and 0.25 to 0.5 % of boracic acid (BA). The mobility of cement and cement-sand slurries is not affected in any substantial way by TA or TABA. Unlike other retarders, these tend to improve the structure of the stone, make it fine-grained, monolithic, and enhance its mechanical strength. At 100 to 120 °C, the strength of the stone declines somewhat after a lapse of two days, owing to a protracted setting of the cement slurry.

Strong retarding properties are common to commercial cream of tartar (CCT), which is a waste product of the food industry. Cream of tartar — potassium tartrate ($KC_4H_5O_6$) — is contained in the juice, bark, and leaves of plants. It forms during fermentation of wines, accumulates in the form of a sediment in fermenters, and deposits in the form of hard crusts on internal surfaces of casks. Commercial cream of tartar is composed of a mixture of potassium tartrate, calcium tartrate, and admixtures.

CCT is capable of effectively retarding the setting time of cement and cement-sand slurries at temperatures of up to 200 °C and pressures of up to 70 MPa.

Insignificant quantities of CCT (up to 0.1 %) at temperatures below 100 °C are apt to accelerate somewhat the setting of cement slurries. CCT, however, ensures an effective retardation of the setting of cement slurries within the entire range of temperatures from 100 up to 200 °C, at pressures of up to 100 MPa. An addition of 0.25 to 0.75 % of boracic acid facilitates the setting retardation both in the case of cement and cement-sand slurries. Large quantities of CCT are poorly soluble. The introduction of CCT helps raise the strength of cement and cement-sand stone hardening at a temperature about 200 °C and pressures up to 70 MPa.

Laboratory Work No. 5

Cement slurry formulation for specified temperature and pressure.

(a) Tests are to be conducted in an autoclave, for example, at a temperature of 75 °C and a pressure of 30 MPa and at a temperature of 120 °C and a pressure of approximately 45 MPa.

Weigh out 300 g of a plugging portland cement and measure out 150 cm^3 of water. Introduce 0.2 % of TA (or any other retarder) into the water. After dissolving TA, pour the water in the bowl containing the cement. After 3 minutes of agitation, pour the slurry into a cup smeared with machine oil. Determine the initial and final set (separately) under the above conditions in the autoclave.

(b) Plugging mixture formulation from a given density (2 g/cm^3) and setting time (the initial set should be 2 hours) at 75 °C (pressure is not considered).

Estimate the amount of water that is to be taken to obtain a slurry with a density of 2 g/cm^3, using a cement of a definite specific surface.

Mix the cement with the necessary amount of water and determine the fluidity of the slurry thus obtained. If this proves to be no less than 18 cm, stop testing and pour the slurry in a cup for determining its setting time; correct the setting time if necessary. Should the fluidity of the slurry prove to be less than 18 cm, add 0.2 to 0.3 % of SAL. This will improve the fluidity of the slurry and make it possible to reduce its water-cement ratio and thus increase its density.

Where the cement requires too much water, introduce coarse-grained quartz sand or some weighting material into the slurry.

After correcting the density and fluidity of the slurry, determine its setting time. Should it prove to be inadequate, increase the content of SAL or use some other setting retarder. Check on the density and fluidity of the slurry once more and then determine its setting time.

(c) Formulation of a plugging mixture with a low water loss and an initial set of 1 hour at 130 °C and a pressure of 45 MPa.

Take cement and bentonite clay in a proportion of 4 : 1 (400 g : 100 g), thoroughly mix them, and add water (approximately 250 cm^3) having CMC dissolved therein in an amount of 0.5 % (by mass). Determine the fluidity of the slurry thus obtained. Should this be below 18 cm, increase the water-cement ratio or introduce 0.2 to 0.3 % of SAL. Having secured the fluidity of the slurry of over 18 cm, verify with the BM-6 apparatus its water loss. If this proves to be within the required limits, estimate (or correct) the setting time of the slurry. In the event of a high water loss, reduce the water-cement ratio or increase the CMC dosage. Thereupon correct the setting time.

Chapter 9

Plugging Cements for High-Temperature Wells

9.1. Cement-Sand Slurries

Most wells are sealed off with pure portland cement slurries. At high temperatures and pressures, slurries made up of a plugging portland cement mingled with quartz sand harden into a long-lasting, durable, impervious stone, resistant to stratal waters.

Under normal conditions, sand added to a slurry of specified setting time is practically an inert component, while at elevated temperatures silica contained in the sand reacts intensively with the cement constituents of the slurry. Quartz sand is found to react not only with calcium hydroxide and hydrosilicates, but also with hydroaluminates, resulting in the formation of hydrogarnet.

Silica, or silicon dioxide, SiO_2, is a mineral most widely occurring in nature. As a component, it is contained in a large group of silicates, clays included. Silica is present in a combined form in cements as well. It is the basic constituent of pozzolan.

At normal temperature, chemically pure quartz is a highly inert substance, but when strongly heated, it reacts with bases. At high temperatures, silica behaves as an acid component, is capable of combining with basic oxides and forming silicates and displacing other acids from their compounds When dissolving in water, quartz reacts with lime while being heated under pressure, and on this principle is based the manufacture of sand-lime bricks. The speed of this reaction depends in a large measure upon the specific surface of the quartz. A perfect crystal weighing 12.8 g, for example, after 48 hours' heating with calcium hydroxyde in an autoclave at 180 °C, loses 0.25 % of its mass. On the other hand, the same crystal crushed down to the size allowing its free passage through a sieve with a mesh size of 0.15 mm loses under similar conditions 12.4 % of its mass. With further grinding into a very fine powder, quartz gets so active that it turns into a colloidal hydrated silica simply after boiling in water.

The mechanical strength of cement stone is found to be influenced not only by the quantity, but also by the nature of the silica additive. Better results have been achieved with additions of ground quartz sand and worse — with additives composed of amorphous-dispersed silica. Under certain conditions of autoclaving, there should be introduced up to 1.5 % of ground sand per each percent of the tricalcium silicate C_3S of portland cement. It is shown that in the course of autoclave hardening of cement stone, much more quartz is being bound than this is necessary for complete interaction with $Ca(OH)_2$, and even somewhat more than is needed for the polybasic hydrosilicates to be converted into monocalcium hydrosilicate. This inference proves the possibility and expediency of introducing increased amounts of quartz sand into plugging portland cements.

The use of fine-grained sand improves the strength of specimens.

Grinding is done with a view to increasing, above all, the specific surface of sand and its reactivity with calcium hydroxide and also raising the activity of some clinker minerals.

As pointed out by academician P. A. Rebinder, the fineness of grinding makes it possible to materially speed up the processes by carrying them out at lower temperatures and under lower pressures. The speed of the processes going on with the participation of substances in the solid state is proportional to the magnitude of the total surface of their particles and, therefore, abruptly increases as they become fractionated.

In view of the fact that in cementing wells there is no need to require that cement stone should have a maximum strength, grinding the sand is not essential. The quartz sand surface reacting with the hydration products of cement can be sifficiently increased by merely increasing the amount of natural-size sand used to prepare the slurry.

In this case, the reaction of calcium hydroxide with silica occurs only on the surface of sand grains, giving rise to the formation thereon of extremely thin (around 5μ) films of gel-like calcium hydrosilicates.

The mechanical strength of cement stone specimens hardened at high temperatures and pressures was found to increase substantially if unground quartz sand, which is readily available in most oil-bearing regions, was added to the plugging portland cement used.

9.2. Choice of Sand

The suitability of sand as an additive to plugging cements should be considered in the light of the changes in the properties of the cement-sand slurry and stone, caused by the addition of sand. Under similar conditions of hydration, the properties of cement-sand slurries are affected to the greatest degree by the mineralogical composition, size, and grain shape of the sand, its pretreatment, etc.

Mineralogical composition of sand. Sand appears as a combination of grains of diverse size, shape, and composition, formed as a result of weathering of rocks. Of most frequent occurrence are quartz sands with an admixture of feldspar grains, mica scales, and of other minerals; found less often are feldspar, limestone, and other sands. Quartz sands are suitable for introduction into plugging portland, slag, and lime cements. Other materials may also be put to use as fillers (aggregates). Ordinary quartz sand contains a high percentage of silica, quite sufficient to bind calcium hydroxide during hydration of cement.

As regards their mode of occurrence, sands are classified into ravine, marine, and river varieties. The last two varieties of sand are, as a rule, of a rounded shape, while the grains of ravine sand are sharply angular. Marine sands, which contain large quantities of organic and easily destructable calcareous admixtures, are not quite fit for use as additives to plugging cements. Most suitable for this purpose are river and ravine sands.

In the general case, an addition of quartz sand to a plugging portland cement is highly desirable, and it is absolutely necessary at high temperatures. At high temperatures, introduction into portland cements of sand, even of a low quality, greatly improves the physical and mechanical properties of cement stone, raises its resistance to the action of aggressive waters, etc. As a general rule, one should make use of a locally occurring sand, resorting to one brought from outside only in exceptional cases.

Sand admixtures. In choosing quartz sands, preference should be given to ones that contain low amounts of the following harmful impurities:

1. Mica, which practically does not combine with cement; it is desirable to use sands containing not more than 0.5 % mica by mass.

2. Sulphates and sulphides, whose quantity calculated in terms of SO_3 should not exceed 1 %. These compounds, while interacting with cement, form hydrosulphoaluminates which in some cases can adversely affect the leak tightness of the cement ring in the annular space.

According to the pertinent USSR standards, quartz sand to be used for addition to portland cement must contain no less than 90 % SiO_2. The presence of small quantities of clay (approximately up to 5 %) increases the density of cement stone without reducing its mechanical strength.

Pretreatment of sand. After extraction, sand should be dried and, preferably, sacked. In working a quarry, it is advisable to flush impure sand, removing dust particles, and then dry it. Sand is to be kept in closed stores. There is no need to calcine sand in order to improve its reactivity.

Granulometric composition of sand. Research into the use of particular cement-sand mixtures should proceed in two directions, namely, (1) the study of the possibility and expediency of utilizing unground sand and (2) the study of the possibility and expediency of utilizing ground sand (obtained by the joint grinding of clinker and sand). The tendency towards utilization of sands of adjusted granulometric composition with a view to ensuring the "rigidity of the matrix" as in the case of concretes cannot be justified, for where well cementing is concerned one should always provide for adequate fluidity of the slurry.

Having assessed the quality of sand as an additive to plugging cements from the standpoint of its granulometric composition, let us point out that any quartz sand is suitable, fine sand carrying a large proportion of dust fractions that would require an increased amount of water being less desirable. It is, however, not warranted to fix the lower boundary for the size of grains, for good results have been achieved even when using ground sand with its specific surface roughly equal to that of cement. With several types of quartz sand available, it is advisable to choose the one in which the fractions passing through a screen with a mesh size of 0.15 mm account for no more than 30 %. Crushing sand is considered inadvisable, but should a need arise for obtaining and applying cement-sand slurries of low density at high temperatures and pressures, then the grinding of sand is mandatory.

Sand carrying a considerable proportion of large-sized fractions is also undesirable, for the latter can precipitate in the receiving tank, under pump valves, etc., thus causing inconveniences in the operation. Proceeding from practical considerations, it is desirable for grains of quartz sand with a density of 2.6 g/cm^3 to be not larger than 0.6 to 0.7 mm, and for sands with a density of 3 g/cm³ and more (quartz-ferruginous sands), not larger than 0.3 to 0.4 mm. Otherwise, sand precipitates during preparation of the cement-sand slurry, which makes it necessary to stir the slurry in the tank.

Cement-sand slurries can be agitated manually, by means of cement mixers, and, at manufacturing plants, by means of grinders, drum mills, mixing screws, and other installations.

The shipment of cement-sand mixtures in cement mixers does not cause any substantial segregation and downward displacement of heavier particles.

9.3. Proportioning of Cement-Sand Slurries

In composing a cement-sand slurry with due regard for hardening conditions, one should take as a guideline the granulometric composition, the content of imprurities and the given amount of sand introduced. For the fluidity of a cement-sand slurry, made in the ratio 1 : 0, to equal 16 to 19 cm, as measured with the standard fluidity test cone, the amount of water required is 38 to 40 % of the cement mass. To obtain a slurry of adequate initial mobility but with a greater quantity of sand (the granulometric composition being constant), the water-cement ratio is to be increased. It would be more proper to recommend that the slurry formulation be selected and adjusted with the aid of the standard fluidity test cone. But this is not always possible under field conditions. Therefore, it is expedient to find a dependence that would allow one to determine an approximate amount of water needed to ensure an acceptable mobility of the slurry at a specified cement-sand ratio, the granulometric composition of sand being known.

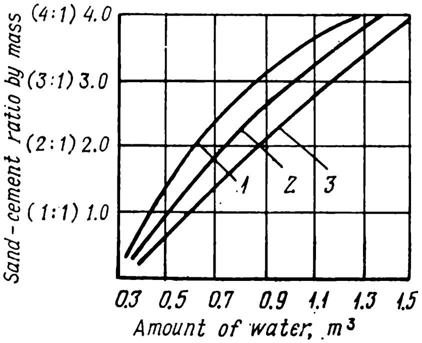

Fig. 75. Graphs for determining the amount of water required to prepare slurries of specified fluidity from cement-sand mixtures differing in sand-cement ratio

Fluidity, cm: *1* — 16; *2* — 17; *3* — 18

It is recommended that the graph of Fig. 75, plotted on the basis of a large number of test results, should be used to determine the necessary amount of water. Bearing in mind that quartz sands occurring in different oil-bearing regions differ in granulometric composition, the tests were conducted with sand 0.3 mm in maximum grain size and having the following granulometric composition:

Sieve mesh size, mm	0.297	0.210	0.149	0.105	0.074	0.053	Pan	Total
Retained on seive, %	1.35	22.20	21.80	14.50	11.70	7.20	21.14	99.89

Referring to Fig. 75, plotted on the Y-axis are sand/cement ratios (by mass) and on the X-axis, the amount of water (in m^3) required for the cement-sand slurry of specified sand/cement ratio to have a fluidity of 16 to 18 cm. With the ratio of the mass of sand of known granulometric composition (comparable with that of tested sands) to the mass of cement being specified, the amount of water required to ensure adequate fluidity of the slurry can easily be found from the curves.

At temperatures of around 20 to 40 °C, a certain amount of sand (up to 30 % in some cases) can be introduced into commercially available plugging cements that yield slurries of increased fluidity. In this case, the resultant cement-sand slurries will have sufficient fluidity, their setting time will remain the same as that of pure cement slurries, while the strength and density of cement-sand stone will be higher than that of pure cement stone, because of reduction in the amount of water retained on the surface of sand grains, and will grow still higher with time. After the lapse of 48 hours, specimens with 30 % of sand, which have been hardening at 22 °C, may not show the strength common to pure cement stone, because of a weak bond between cement and sand grains. An increase in the dosage of sand requires raising the water-cement ratio and this brings about a reduction in the strength of cement-sand stone.

Raising the ambient temperature to 75 °C enables one to increase the amount of quartz sand in the slurry up to 50 % of the mass of cement without impairing the strength of two-day old specimens. Under such conditions, as the specimens age, sand starts playing a positive role, binding a certain amount of calcium hydroxyde whose presence lowers the strength of cement stone.

With the ambient temperature raised to 110 °C, the physical and mechanical properties of cement-sand slurries change more drastically.

Table 27 lists comparative data on pure cement and cement-sand specimens hardening under different temperatures and pressures.

At a temperature of 140 °C and pressure of up to 40 MPa, the specimens show a considerably higher mechanical strength. Under still more severe hardening conditions ($T = 200$ °C, $p = 50$ MPa), the mechanical strength of the specimens also changes, reaching its maximum in the specimens having their cement/sand ratio close to 1 : 1.

The fracture surfaces of cement-sand specimens hardened at high temperatures (140 to 200 °C) and pressures, when viewed under not very high a magnification, show that individual quartz

Mechanical Strength of Cement-Sand Specimens with Progressively Increasing Sand Content as a Function of Ambient Temperature and Pressure

Mixture composition	24-h strength, MPa						48-h strength, MPa					
	$T - 110$ °C $p - 30$ MPa		$T = 140$ °C $p = 40$ MPa		$T = 200$ °C $p = 50$ MPa		$T = 110$ °C $p = 30$ MPa		$T = 140$ °C $p = 40$ MPa		$T = 200$ °C $p = 50$ MPa	
	bending	compression	bending	compression	bending	compression	bending	compression	bending	compression	bending	compression
1 : 0	—	—	1.96	5.0	—	—	4.38	16.7	1.97	5.0	1.91	4.7
2 : 1	4.20	—	8.85	27.9	10.13	36.8	4.55	18.7	8.85	29.2	11.11	36.7
1 : 1	—	—	—	—	—	—	4.48	—	9.00	30.6	—	—
1 : 2	2.27	6.6	8.00	28.0	6.60	22.0	4.28	11.7	8.60	28.2	6.43	24.4
1 : 3	—	—	—	—	—	—	2.50	7.3	6.56	20.7	4.82	17.0
1 : 5	—	—	4.9	—	—	—	—	—	5.69	13.3	4.04	12.7

sand grains have a rough, pitted surface providing for a much stronger bond with the binder than that existing in the bulk of pure cement stone.

The advantages of cement-sand slurries over pure cement ones are quite obvious and therefore they should be recommended for universal use in cementing deep, high-temperature wells.

At high temperatures (above 100 °C) and pressures, the introduction of quartz sand into plugging portland cements helps obtain a cement stone exhibiting better physical and mechanical properties than that resulting from slurries prepared with an amorphous silica (opoka).

9.4. Permeability of Cement-Sand Stone

Introduction of quartz sand into portland cements that harden at high temperatures and pressures appreciably augments the density of cement-sand stone (Table 28).

Table 28

Permeability of Two-Day Old Cement and Cement-Sand Stone Specimens as a Function of Sand Content and Hardening Conditions (Karadag Cement)

Hardening conditions		Permeability, μ^2, of specimens with cement/sand ratio of					
T °C	p, MPa	1 : 0	2 : 1	1 : 1	1 : 2	1 : 3	1 : 5
110	30	0.27	0.58	1.35	—	—	26.00
140	40	29.60	0.00	0.00	0.00	0.00	0.00
200	50	30.40	0.00	0.00	0.00	0.00	0.00

Where the maximum grain size of sand is from 0.2 to 0.3 mm and no plasticizer is used, it is advisable to utilize cement-sand mixtures containing up to 100 % sand. At normal temperature, the decrease in the permeability of cement-sand specimens is more pronounced, but requires a longer period of hardening.

The physical essence of the increased mechanical strength and reduced permeability of cement stone containing quartz sand and hardening at high temperatures and pressures is to be sought in the chemical interaction of silica with free lime and other products of hydration of clinker, in the improved structure of the stone, in the replacement of cement particles of low strength by high-strength quartz, etc. With mounting temperature and an increased proportion of quartz sand (till a certain limit), these factors become increasingly active.

Since in cementing wells cement is, as a rule, used in its pure form, additions of quartz sand help to appreciably curtail the cost of wells.

The application of cement-sand mixtures for cementing deep, high-temperature wells makes indispensable the treatment of these mixtures with setting retarders. The composition of additives is selected in a manner analogous to the choice of a formulation for preparation of portland cement slurries without sand.

9.5. Slag-Sand Cements

In slag-sand cements, the role of the binder is played by slag, whose activity increases with rising temperature, and that of the setting retarder and active component, by quartz or quartz-magnetite sand.

Blast-furnace slags, long since employed in the construction industry, closely approach by their chemical composition the portland cement clinker, usually differing from it in a lesser proportion of calcium oxide. To intensify the processes of setting and hardening, slags are normally ground, for the purpose of enrichment, together with lime and portland cement clinker. Lime-slag, slag portland, and gypsum-slag cements have been developed and found wide application in the construction industry.

Millions of tons of slag are obtained during smelting of metals as a melt of non-metal minerals, always contained in metal ores, and of fluxes that are added to impart a degree of fusibility to the slag and to clear from the liquid metal undesirable impurities (phosphorus, sulphur, manganese). Smelting of 1 t of pig-iron is attended by the formation of approximately 1 t of slag.

When granulated, i. e., after quick cooling in special granulating units, blast-furnace slags are used in the production of a wide variety of cements. Presently, the Soviet construction industry has at its command several different types of binding materials with blast-furnace slag as their chief component. Blast-furnace slags, both basic and acid, are also used, mainly in granulated form, in the cement industry.

Blast-furnace slag, a non-metallic product that consists mainly of silicates and calcium aluminates, is obtained in the blast-furnace together with iron in the form of a melt which is subsequently cooled. A rapid cooling with water, steam, or air forms granulated slag, while with a slow cooling, there emerges lump one. Blast-furnace slag becomes highly active hydraulically on its being very quickly cooled or granulated with water. A slowly cooled slag has enough time to become crystallized to some degree and then its hydraulic properties decline. A slag granulated with a small amount of water (so-called dry-granulation slag) displays an insignificant activity. As such, slag, even if granulated, is normally little active when mixed with water, and so an addition of accelerators is necessary to activate it. But with mounting temperature and pressure, the activity of slag increases to such an extent that frequently it should be used with addition of setting retarders.

The chemical and mineralogical composition of slag and the method of its cooling are factors determining the physical and mechanical characteristics of slag cement, *viz.*, the setting time, mobility, mechanical strength, etc. Alumina (Al_2O_3) is considered to be the most valuable constituent of slags. Silica (SiO_2) lowers the hydraulic properties of slags, and its elevated level may be warranted only by the fact that during granulation it is easier to obtain a vitreous mass from the slags, this mass being an indispensable component for adding hydraulic properties to slags. Magnesia (MgO) has a positive effect on the hydraulic characteristics and replaces lime. It is believed that an excess amount of MgO can cause an enlargement of cement stone volume. Phosphorous oxide (P_2O_5) is held to be a harmful admixture, though this has not as yet been definitely established. Manganous oxide (MnO) is also referred to harmful admixtures, the ones that depress the reactivity of slags. It has been, however, found that at very high temperatures and pressures slag cements with an elevated content of MnO are apt to set at a slower rate. The USSR standard GOST 3476-74 stipulates the following proportions of manganous oxide in the acid blast-furnace slags intended for the production of construction cements: no more than 2 % for Grade I slags and no more than 4 % for Grade II slags; the respective figures for basic slags are 2 and 5 %.

When present in small quantities, ferrous oxide (FeO) does not affect the mechanical strength of cement stone. But an increase of its content up to 10 % and more results in "ferrous disintegration" of slag, caused by ferrous oxide turning into ferric oxide and its subsequent volumetric expansion. This circumstance cannot apparently be regarded as a negative factor in cementing casing strings. Sulphides are counted among harmful admixtures and their quantity is strictly limited by the aforementioned standard. All of the basic slags should not contain more than 3.6 per cent of it. It is common knowledge that more active cements are obtained from granulated slags, but, eventually, their activity declines at a greater pace than in the case of lump slags.

In obtaining plugging cements, use may be made of both granulated and lump slags, but preference is to be given to the former, especially with temperatures reaching 150 to 170 °C.

In each concrete case, the choice of slags and evaluation of their properties with special reference to cementing of oil wells should be made on the grounds of laboratory assays.

It is believed that the higher the activity index I_a ($I_a = Al_2O_3/SiO_2$) of slags, the better their hydraulic properties and the stronger the stone they yield.

USSR standard GOST 3476-74 stipulates that the activity index should be no less than 0.17 for basic slags and no less than 0.33 for acid ones.

According to the above standard, it is recommended that the quality of slags should also be checked with reference to their basicity index I_b $[I_b = (CaO)(MgO)/(SiO_2)(Al_2O_3)]$ which should be no less than 1.0 for basic slags and no less than 0.65 for acid slags.

But in many instances granulated basic slags, while being of a similar chemical composition, yield mixtures widely differing in their plugging characteristics. Of prime importance in obtaining plugging cements are the degree and mode of granulation, storage conditions, temperature of the slag, and many other as yet unknown factors.

Most varieties of granulated blast-furnace slag differ but little in their density, all of them being lighter than portland cement.

The properties of the slag cements differ substantially from those of portland cement. Normally, slag cements harden at a very slow rate, but with the ambient temperature rising to 100 °C and higher, the processes of setting and hardening gain in intensity and slag-sand cements form a dense and strong stone that is highly resistant to aggressive media. The other technical characteristics, such as volumetric changes, the need for water in mixing, mobility, etc., closely approach those of portland cement. Their elevated resistance to attack by mineralized and fresh waters justifies recommending slag cements for use as a plugging material in oil and gas wells, where, as is known, the presence of highly mineralized water adversely affects the durability of portland cement stone.

9.6. Setting Time and Mechanical Strength of Slag-Sand Slurries and Stone

Most slurries prepared with granulated blast-furnace slags as their principal component become set and harden into a stone both at high and low temperatures. At room temperature and atmospheric pressure, their setting is greatly retarded, and it takes a long period of time for the strength of the stone they yield to become adequate, whereas at elevated temperatures these processes occur more rapidly. Thus, a slag-sand slurry with a slag/sand ratio of 1 : 1 and enough water for it to show a fluidity of 16 to 17 cm in the standard fluidity cone test remains fluid for more than a month. Its setting and hardening into stone 15 to 20 MPa in compressive strength require from 5 to 6 months. While in the process of hardening, the slurry does not suffer segregation. Sand, even one with as high a density as 3.07 g/cm^3, does not settle, although the slurry remains fluid for a long time.

The activity of slag-sand slurries grows higher with increase of temperature. Some kinds of slag get set in 1.5 to 3 hours right after having been ground, when tested in water at 75 °C, which shows that they can be used in wells with this temperature. Unlike portland cement, slag-sand one is not susceptible to an intensive thickening for quite a long time after having been mixed with water.

Under ordinary conditions, the gel strength of slag slurries is not great and changes but slightly over a space of 30 minutes

The setting time of slag-sand slurries grows longer with increase of their sand content, and the greater the sand content, the more intensive is the setting retardation, so that a slurry with a slag/sand ratio of 1 : 1 requires 15 times more time to set than a pure slag slurry, no matter what the kind of the sand used. Quartz-magnetite sand retards the setting of slag cements somewhat more than pure quartz sand. With greater amounts of sand in the mix (up to 30-40 %) a gain in strength is observable. Should the proportion of sand be raised still more, the strength declines with the use of both kinds of sand. At 90 °C, the permeability of test pieces is close to zero, whereas the permeability of specimens made of pure slag reaches $(10 \text{ to } 15) \times 10^{-3}\ \mu^2$.

Increasing temperature to 100-130 °C tends to accelerate the setting rates and raise the mechanical strength.

The setting time of slurries hardening at 110 to 125 °C is much shorter than at 75-90 °C, and if no sand is added to them, they often prove unsuitable for cementing wells.

With higher temperatures and pressures, the setting of slurries prepared of pure, freshly ground granulated slags takes, as a rule, much less time, and when used for cementing wells, they should be mixed with sand.

The mechanical strength of specimens also depends on the amount of sand introduced. All two-day old slag-sand specimens are stronger than are specimens of pure slag. The strength of slag-sand specimens is the highest with addition of 20 to 40 % of sand.

To give a general characteristic of a large quantity of granulated blast-furnace slags, stored after having been ground for a period of 1 to 9 months, a graph (Fig. 76) was plotted, showing the relationship between the setting time slurries, made with different types of slag, and the sand dosage. The curves, plotted on the basis of about 700 assays, present a qualitative valuation of slag-sand mixtures as plugging slurries in general and help form a judgement on their possible use at a temperature of 150 °C and a pressure of 50 MPa. It is seen from the graphs of Fig. 76 that a higher percentage of sand in the mixture leads to a longer period of setting.

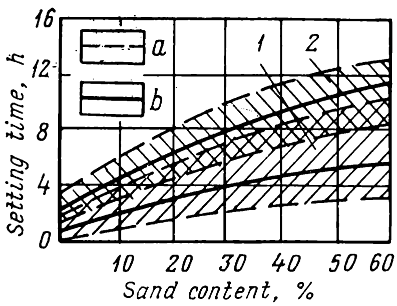

Fig. 76. Effect of sand content on the setting time of slag-sand slurries (t = 170 °C; p = 50 MPa)

(*a*) initial and final set ranges; (*b*) averaged values; *1* — initial set; *2* — final set

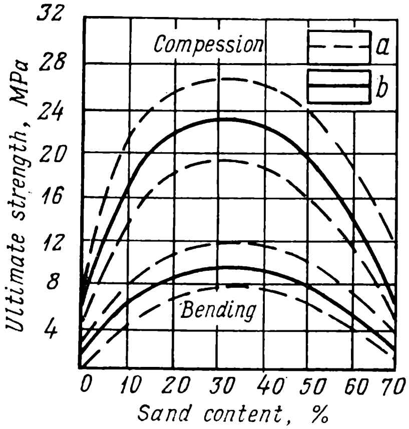

Fig. 77. Effect of sand content on the mechanical strength of slag-sand stone (t = 150 °C; p = 50 MPa)

(*a*) compressive and bending strength ranges; (*b*) averaged values

Figure 77 shows curves, plotted on the basis of experimental data, which establish changes in the bending and compression strength of two-day old slag-sand test pieces containing different proportions of sand at 150 °C and a pressure of 50 MPa. Here, use was made of the same types of slag as in establishing the setting rates. Sand tends to intensively heighten the strength, and with 20 to 40 % of it in the mixture the strength of two-day old specimens reaches its maximum. Subsequent addition of sand downgrades the strength of stone.

To obtain slag-sand slurries with setting rates acceptable in the practice of cementing deep wells with temperatures as high as 200 °C and pressures of 50 to 70 MPa (Fig. 78), the quantity of sand should be increased to 30-70 % by mass. After grinding, the slags were stored for 1 to 9 months.

The mechanical strength of a two-day old slag-sand stone changes at 200 °C and a pressure of 50 MPa in the same manner as the strength of specimens at 150 °C and pressure of 50 MPa, but its maximum shifts somewhat towards an increased dosage of sand (Fig. 79). Individual slag-sand test pieces that hardened at 200 °C and a pressure of 70 MPa show a bending strength of 12 to 14 MPa, their strength in compression reaching 30 MPa. In all cases involving the use of slag-sand cement with any proportions of slag and sand (except for 1 : 0), the permeability of one-, two-, and seven-day old specimens was found to be close to zero. Test pieces recovered from a well, where they hardened for 60 hours at 140 °C and a pressure of around 40 MPa proved impervious as well.

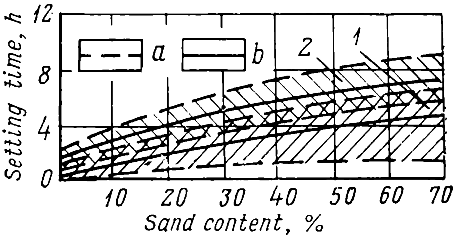

Fig. 78. Effect of sand content on the setting time of slag-sand slurries ($t = 200\,°C$; $p = 50$ MPa)

(*a*) initial and final set ranges; (*b*) averaged values; *1* — initial set; *2* — final set

An excess over the optimal quantity of sand tends to reduce the mechanical strength of stone, for then the active constituents are insufficient to provide for complete binding of ground sand.

The introduction of unground sand also improves the strength of slag-sand stone, though in some cases the absolute value of the strength increment is somewhat less. Considering the much smaller specific surface of unground quartz sand, the quantity of the sand may be increased.

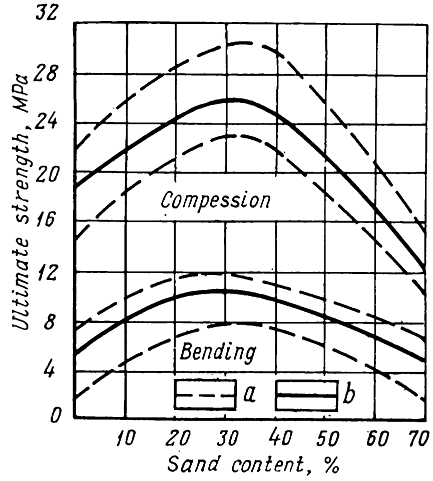

Fig. 79. Effect of sand content on the mechanical strength of slag-sand stone ($t = 200\,°C$; $p = 50$ MPa)

(*a*) compressive and bending strength ranges; (*b*) averaged values

The higher the ambient temperature, the greater the extent of hydration of slag in itself. Variations in the degree of hydration of slag constituents can be traced by following changes in the density and porosity of the hardened slurry. As the temperature increases from 110 to 200 °C at a pressure of 40 MPa, the density of pure slag slurries drops from 2.66-2.68 to 2.46-2.48 g/cm^3, and so does their porosity, this pointing to the fact that the extent of hydration of slag grains grows higher. At 200 °C, the porosity curve starts to flatten out, and so does the density curve. This means that either a higher temperature or a longer hardening period is necessary for the slag grains to hydrate completely.

9.7. Slag-Sand Cements for Wells with Bottom-Hole Temperatures Above 200 °C and Pressures up to 100 MPa

For cementing wells with a bottom-hole temperature of over 200 °C and pressures of up to 100 MPa mixtures containing acid blast-furnace slags as their main component have proven most promising. They display acceptable setting rates and yield a dense, shrink-proof stone of quite a sufficient strength. The test pieces showed it to be absolutely gas-tight.

The setting time of slurries prepared with acid slags grows with increasing proportion of quartz-magnetite sand in them. Thus, a slag-sand ratio of 1 : 1 by mass ensures obtaining slurries with sufficient setting rates. Such slurries may be put to use at higher temperatures and with greater pressures. Thus, even at temperatures of 250 to 300 °C and pressures as high as 100 MPa one may compose mixes with blast-furnace slags as the principal component that can possess qualities needed for cementing and obtain therefrom an impervious stone of a sufficiently high strength.

The handling of other types of slag demonstrated that these too may be utilized in cementing deep, high-temperature wells, but the formulation of the slurries should be composed for each concrete case. Slags that fail to yield slurries with long setting times in conditions under review shortly after grinding, become slow-setting when kept stored for a certain time.

9.8. Slag-Sand Cements with Sand of Natural Size

One of the advantages of the slag-sand cement obtained through intermingling milled slag and sand is its comparatively low cost, the principal item being the cost of grinding. These expenditures can be brought down by employing quartz of quartz-magnetite sand of natural size. In the case of freshly-ground slags, this does not result in any noticeable deterioration of the quality of either stone or slurry. Such slurries are characterized by a certain acceleration of setting, as compared to ones which carry additions of ground sand.

In this case, for use are fit various kinds of quartz sand, starting from desert and ending with river varieties.

The setting rates of slag-sand slurries can be controlled within desirable limits through introduction of sand. The mechanical strength of cement stone is determined largely by the action of temperature. The permeability of stone is close to zero.

As an example, we may adduce the results of testing the slag of the Zhdanov smelter and quartz sand.

At a given temperature, the greater the age of the slag stone, the higher its mechanical strength. Thus, at a temperature of 130 °C and pressure of 40 MPa, the bending strength of pure slag stone was found to be 1.5 MPa for two-day old specimens, 3 MPa for seven-day old specimens, and 3.2 MPa for fourteen-day old specimens. The compressive strength of the specimens behaved in a similar manner.

The introduction of quartz sand helped raise the absolute values of the ultimate strength in bending and compression, but the character of the changes remained the same. Judging by variations in the mechanical strength of specimens, most desirable seem to be slag/sand ratios of 2 : 1 and 1 : 1.

Slag-sand specimens of all ages that hardened at a temperature of 170 °C showed much higher absolute values of mechanical strength than those hardened at 130 °C. The mechanical strength of pure slag stone, which hardened at a temperature of 170 °C and pressure of 40 MPa, increased with age. Thus, the bending strength values were found to be 2.2 MPa for two-day old specimens, 5 MPa for seven-day old specimens, and 5.7 MPa for fourteen-day old ones. The introduction of 33 % (by mass) of sand (slag/sand ratio 1 : 3) increased the strength of two-day old specimens to 7 MPa.

The strength of the specimens was also found to rise with age, as well as with sand content increasing up to the ratio 1 : 1.

The permeability of both pure slag and slag-sand specimens hardened for 2, 7, and 14 days at a temperature of 170 °C and pressure of 40 MPa was found to be no more than 2×10^{-3} μ^2. The stone specimens showed very dense, fine-grained structure and were found to be practically impervious to water.

A slight magnification allows one to discern a very dense structure of slag-sand stone that has been hardening at high temperatures and pressures. The bond between sand grains and the slag constituent is extremely great, this being corroborated by fractures on individual quartz grains.

But in some cases the stability of slag-sand slurries proves insufficient and their setting time (especially with the use of freshly ground slags) is short, with the density of the slurries showing a substantial increase.

Slag-sand slurries are treated with setting retarders, if required.

9.9. Plugging Cements Based on Ferromanganese Slag

Ferromanganese slag contains from 5 to 12 % manganous oxide, which makes it practically incapable of setting and hardening under ordinary conditions. At temperatures over 100 °C and high pressures, ground granulated ferromanganese slags begin to exhibit binding properties. They may be utilized as a binding material at temperatures of 150 to 300 °C. The upper limit for the use of ferromanganese slags is, evidently, the temperature range from 350 to 400 °C.

At a temperature of 175 °C and pressure of 70 MPa, the initial set of a chemically untreated ferromanganese slag-sand slurry takes 4.5 to 5.5 hours and at a temperature of 200 °C and pressure of 100 MPa, it takes more than 3 hours.

Should it become necessary to use ferromanganese slag at lower temperatures, it has to be either treated with setting accelerators, or ordinary blast-furnace slag should be added to it. A strong accelerator for this type of slag is soda ash, whose introduction in an amount of 1 % of the mass of slag permits of obtaining slurries with an initial set of 2 to 2.5 hours at a temperature of 125 °C and pressure of 40 MPa. The application field of ferromanganese slag can be extended by adding to it conversion pig-iron slag.

9.10. Jointly Ground Slag-Sand Cements

The greatest effect in retarding the setting of slag-sand slurries is ensured with joint grinding of slag and quartz sand. If, all other things being equal, the initial set of a slag-sand slurry using unground quartz sand occurs in 1 hour, the use of ground sand prolongs this time approximately to 1 hour 30 minutes. The use of jointly ground slag-sand cement helps prolong the initial set to 4 hours.

The effect of retarding the setting of slurries made of jointly ground slag-sand cements is observed to take place in the range of temperatures from 200 to 250 °C and pressures from 100 to 120 MPa. The retarding power of sand crushed together with slag is typical of all kinds of quartz sand containing no accelerating admixtures.

The setting time of slurries made of slag-sand cement is affected not so much by the fineness of the grinding, but rather by the method of grinding.

The mechanism of retarding the setting (structurization processes) of slag-sand slurries of jointly ground slag and sand consists in changing the surface properties of the powdered sand and slag. Due to their active centres, the minute slag particles become adsorbed on the amorphous surface of quartz. Amorphous silica can be adsorbed on large slag granules. As a result, the most soluble (most active) centres of slag particles remain for some time isolated from the action of water. The dissolution rate of slag components diminishes, and this leads to the lengthening of the setting time.

9.11. Separate and Combined Effects of Temperature and Pressure on Properties of Slag Slurries

Typical of all kinds of slag is the acceleration of their setting with rising temperature at a constant pressure.

The mechanical strength of two-day old specimens increases steadily with temperature.

The effect of pressure on slags of different origin is dissimilar and is strongly influenced by the temperature at which the slurry is maturing and the time and conditions of slag storage after grinding.

With an increase of pressure, but at a constant temperature which favours a rapid setting of the slag-sand slurry, the initial set period gets shorter (Fig. 80). The strength of two-day old slag-sand stone specimens at first grows somewhat and then drops slightly, but, generally, the rise of pressure has no significant effect on the strength of the specimens. At temperatures of up to 200 °C, increasing the specific surface of slag improves the strength of two-day old slag specimens.

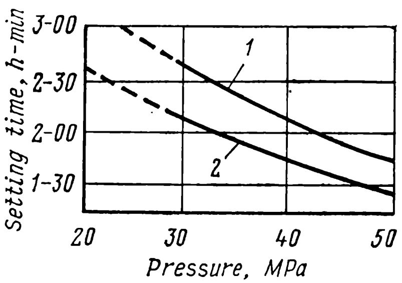

Fig. 80. Effect of pressure on the setting time of a slag slurry (1 : 0) at a temperature of 100 °C

1 — initial set; 2 — final set

9.12. Effect of Storage Time on Properties of Slag Cements

The characteristics of cements containing blast-furnace slags as their major component are apt to change during their protracted storage after grinding, and this is to be taken account of in selecting the composition and ascertaining the suitability of a given mixture in concrete conditions. The storage of unground slag (for up to 1 year) has almost no effect on the properties of the slurry prepared therefrom. The properties of ground slag change more significantly with longer storage time and higher atmospheric moisture; its water requirement increases too, while the setting becomes retarded. The mechanical strength of test pieces changes but slightly.

In certain circumstances, the ability of slags to prolong their setting time upon storage after grinding may be considered a positive factor, and it should be taken advantage of in preparing mixtures for use in deep, high-temperature wells. Keeping slag in an exsiccator during the first 4 to 6 months produces but little practical effect on its properties. The most substantial change in the setting time of slag slurries occurs initially after grinding the slag (Fig. 81). It has been noticed that the finer the slag is ground, the more intensively its activity falls.

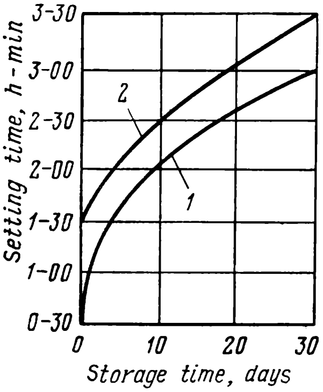

Fig. 81. Effect of slag storage on the setting time of slag slurry ($t = 140\,^\circ C$; $p = 40$ MPa)
1 — initial set; 2 — final set

The viscosity of slag slurries increases maximally during the first one-two months of slag storage. At lowered temperatures, the greatest retardation in the setting of the slurries was found to occur in the first two-four months of slag storage. With rising ambient temperature and pressure, the setting rate slows down less intensively.

Depending upon their period of storage, ground slags kept in open containers in a shed change the properties of slurries prepared therefrom in approximately the same manner as in the case of their being kept in closed storage rooms, but their activity in the first case declines more markedly.

9.13. Water Loss of Slag Slurries

One of the most essential disadvantages of slag-sand slurries is quick separation of water. The characteristics of the slag-sand slurries can be improved by introducing into them some bentonitic clay.

As the amount of bentonitic clay is increased, the amount of mixing water required to obtain a slurry of adequate fluidity (17 to 18 cm) grows larger, reaching 53.4 % at a clay content of 12.5 %, whereas the pure slurry requires only 33.5 % of water to have the same fluidity. Increasing the clay content reduces the water loss of the slurry, so that eventually, with the amount of clay reaching a certain value, it becomes practically zero.

A slag-sand slurry with approximately one third water loses most of it through filtration in the first few minutes after mixing. Introducing into the slurry small quantities of bentonitic clay helps reduce its filter loss, so that even as low a clay content as 5 % by mass of dry mixture nearly halves the amount of water lost through filtration. With 12.5 % of bentonitic clay in the slurry, its filter loss amounts to 0.3 % in 5 min, 3 % in 30 min, and 6 %

in one hour, although its water content has been increased to provide for wetting the clay.

Also, introduction of bentonitic clay into slag-sand slurries helps reduce their density.

9.14. Adhesion of Slag Cements to Metal

The adhesion of slag-sand cements to a steel rod depends upon temperature, pressure, water-slag ratio, and other factors.

Figure 82 illustrates changes occurring in the bond between slag and slag-sand slurries and a steel rod with rising temperature. As evidenced from the diagram, the bond between a maturing slag-sand slurry and metal grows stronger with temperature and time.

Application of a clay mud to the surface of steel rods always results in reducing the bond down to zero. In pulling variable cross-section rods from an encased cement stone, the bond steeply increases, and the total load reaches high values, which shows that the encased slag-sand stone is in compression.

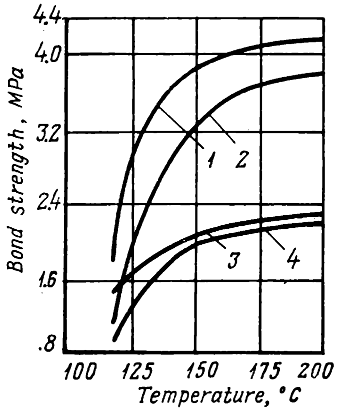

Fig. 82. Effect of temperature on the adhesion of slag and slag-sand stone to a steel rod

Kind and age of specimens: 1 — slag, t = 48 h; 2 — slag, t = 96 h; 3 — slag-sand (2.3 : 1), t = 48 h; 4 — slag-sand (2.3 : 1), t = 96 h

9.15. Slag Portland Cements

To make successful use of slag and slag-sand cements at temperatures below 100-120 °C, an addition of hardening intensifiers, such as portland cement, is indispensable. Table 29 lists data on the setting time of slag slurries containing different proportions of plugging portland cement.

It is seen from the figures in Table 29 that at 22 °C pure slag slurry sets very slowly. An addition of 10 % of portland cement markedly reduces the setting time of the slurry, portland cement playing the principal part in this. But any further increase in the proportion of portland cement produces merely a slight successive acceleration of the setting rates and, therefore, it appears inadvisable to use larger quantities of it. With the ambient temperature going up to 75 °C, the activity of slag grows higher, and already an addition of 3 % of portland cement brings about a very pronounced shortening of the setting time, this being mainly due to the part played by portland cement in speeding up the setting rate: the quicker the portland cement sets, the more intensive is the setting of the slurry.

Table 29

Setting Time of Slag Portland Slurries at 22 °C

Slurry composition, % by mass			Setting time, h-min	
Slag	Cement	Water	Initial set	Final set
100	0	29	60—00	64-00
90	10	29	6—50	8-00
80	20	29	6—50	7-40
70	30	30	6—40	7-40
50	50	30	6—00	7-10
30	70	46	5—25	6-50
10	90	40	5—00	6-35
0	100	40	5—00	6-35

The mechanical strength of slurries of the above composition varies as a function of the quantity of portland cement introduced. Raising the proportion of the latter from 3 up to 17 % of the mass of the mix increases the bending (compression) strength 3.2 (6.2) times in one day, 2.6 (6.2) times in two days, and as much as 3.4 (7.2) times in 7 days. To obtain a stone with higher strength characteristics, it is expedient to introduce 10 to 20 % of portland cement. The setting rate of such slurries will be somewhat slower than in the case of slurries made of portland cement alone. With longer maturing of the former, their ultimate strength rises both in bending and compression. With an increased proportion of portland cement, the permeability of slag portland cement specimens diminishes. The density of slag portland cement stone increases also with a longer period of the specimens' hardening.

Any further increase in temperature and pressure brings with it an intensified maturing of slag slurries. When the bottom-hole temperature exceeds 80 to 90 °C, slag portland cement slurries cannot be put to use without preliminary laboratory assays. The setting rates of these slurries are determined mainly by those of portland cement, and, as a rule, the setting starts and goes on at a quick pace, which precludes recommending their use for cementing wells without an additional chemical treatment. Provided the composition of a slag slurry has been selected properly and with due regard for the slag storage time, these slurries may be safely employed at 100 to 125 °C without addition of activators.

The mechanical strength of slag portland cement specimens hardening at 75 °C grows higher with age, provided that no more than 60 % of portland cement is introduced into the slurry (Table 30).

Maturing of the specimens at 75 °C with the proportion of portland cement exceeding 60 % results in a slight weakening

Table 30

Physical and Mechanical Properties of Slag Portland Cement Specimens Hardening at 75 °C

Slurry composition, % by mass			Specimen strength, MPa			
			2-day old specimens		10-day old specimens	
slag	portland cement	water	in bending	in compression	in bending	in compression
100	0	42	—	—	2.4	8.5
90	10	42	2.3	4.7	2.7	11.5
80	20	43	3.5	15.2	5.2	16.0
70	30	44	4.6	16.9	5.6	20.2
60	40	44	5.3	20.9	6.0	20.7
50	50	43	5.9	22.1	6.3	22.1
40	60	42	6.0	22.3	6.9	23.0
30	70	41	7.4	27.9	7.2	—
20	80	40	8.9	30.9	8.1	35.5
10	90	38	8.9	37.4	6.3	30.2
0	100	35	10.0	41.9	—	—

of their strength by the 10th day. Contamination of slag-sand cements with portland cement and failure to take account of this during chemical treatment lead to a sharp acceleration of the setting rate.

Other materials, such as lime and gypsum, may also be used as setting accelerators for slag-sand slurries. The use of lime for controlling the setting time of slag mixtures enables one to obtain slurries with prolonged setting but reduced mechanical strength.

9.16. Lime-Sand Slurries

Lime-sand slurries can be obtained by mingling together quicklime (air-slaked lime and lime paste), ground quartz sand, and bentonitic clay to stabilize the slurry. The slurries get set and harden into stone at high temperatures. At low temperatures (below 100 °C) the slurries remain pasty for a long time. Higher temperatures and pressures lead to a drastic acceleration of the setting rates. At temperatures of 130 to 150 °C and a pressure of 50 MPa, the initial set of

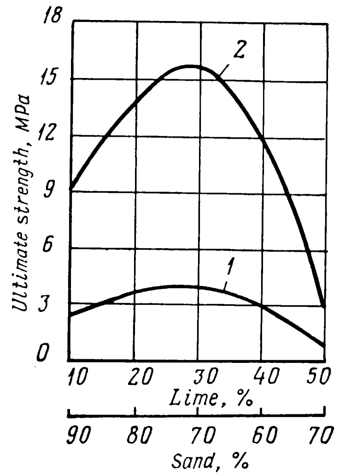

Fig. 83. Mechanical strength of two-day old lime-sand stone (t = 150 °C; p = 50 MPa)

1 — bending strength; 2 — compressive strength

the slurries takes less than 30 minutes. Tartaric acid (1.25 %) in combination with boracic acid (0.25 to 0.50 %) performs the role of a retarder. This quantity of the retarder provides for obtaining a lime-sand slurry with an initial set period of 1.5 to 2 hours at temperatures of 150 to 200 °C and a pressure of 50 MPa.

Under these conditions, the strength of two-day old stone is roughly 3 to 5 MPa in bending and 10 to 20 MPa in compression (Fig. 83). The permeability of the stone approaches zero.

In place of quartz sand, use may be made of crushed silica additives, such as trass, tuff, pumice, diatomite, volcanic ash, and others.

9.17. Belite-Silica Cement

Belite-silica cement (BSC) has been specially developed for cementing deep, high-temperature wells. It is obtained by jointly grinding the waste products of extraction of alumina, soda, and potash from nepheline ores and also quartz sand. The binder here is nepheline, or belite, sludge. The bulk (up to 80 %) of nepheline sludge is constituted by β-$2CaO \cdot SiO_2$. The total amount of hydrated grains ranges from 5 to 7 %.

Under normal conditions, nepheline sludges harden very slowly. Their activity is influenced by the temperature at which they are being dried and also by the presence of activators (lime, gypsum, portland cement).

The setting rates of belite-silica slurries grow higher with rising temperature and pressure, and so does the mechanical strength of the stone they yield. The manufacture of BSC consists in drying and joint grinding of the constituents. The cement constituents may also be ground separately and then mixed together.

The setting time of BSC slurries and the mechanical strength of the stone they turn into depend on the slurry composition, the specific surface of the constituents, hardening temperature, and the amount of water in the slurry. The optimum silica content is from 30 to 50 %. At temperatures from 160 to 200 °C and pressures from 60 to 70 MPa, the initial set of such a slurry occurs in 1.5 to 4.5 hours after mixing. When stored for a long time, especially in conditions of high humidity, belite-silica cement gets lumpy and cakes, but to a lesser extent than portland cement. Introduction of portland cement accelerates the setting of belite-silica slurries.

Practical Studies

Calculation of cement-sand mixture.

The mass P of a cement-sand mixture may be calculated by the formula

$$P = C(A + B + 1) \tag{41}$$

The volume V of the slurry obtained from the above amount of the mixture is found from the formula

$$V = C/\rho_c + A + B/\rho_s \tag{42}$$

and its density, from the formula

$$\rho = \frac{A + B + 1}{1/\rho_c + A + B/\rho_s} \tag{43}$$

where C is the mass of cement, ρ_c is the density of cement (for portland cement $\rho_c = 3.12$ to 3.15 g/cm^3), ρ_s is the density of sand (for quartz sand $\rho_s = 2.65$ to 2.75 g/cm^3 and for quartz-magnetite sand $\rho_s = 3.05$ to 3.08 g/cm^3), A is the water-cement ratio, and B is the sand-cement ratio.

With $W : C = A$ and $S : C = B$, the volume V_1 of the slurry obtained from 1 ton of the cement-sand mixture may be calculated by the formula

$$V_1 = (1 - BC)/\rho_c + BC/\rho_s + A\,(1 - BC) \tag{44}$$

and the volume V_2 of the slurry obtained from 1 ton of cement, by the formula

$$V_2 = 1/\rho_c + B/\rho_s + A \tag{45}$$

When using cement-sand mixtures with sand/cement ratios greater than 2 : 1, one should increase A to 0.6 with reference to the curves of Fig. 75.

Laboratory Work No. 6

Following directions contained in the section "Proportioning of Cement-Sand Mixture" of the present chapter, compose cement sand slurries differing in the content of sand and measure their density.

Chapter 10

Cements for Low-Density Slurries and Weighted Cements

In the general case, an increase in the depth of wells results in that the interval needed to separate beds becomes longer and in the vertical section there appear a large number of horizons that have to be sealed off. Penetration of gas into the annular space and further on to the wellhead is of frequent occurrence consequent upon the use of casing pipes with standard threads and ordinary plugging cements in developing gas and gas-condensate fields.

In order to lessen the possibility of gas movement, the plugging mixture must be brought to a great height, right up to the wellhead, to the shoe of the preceding casing string, or to the level of compact rocks, ensuring at the same time the formation of cement stone of adequate density (of minimal permeability). By filling the annulus over a considerable height with some dense material it becomes possible to increase the hydraulic fracturing pressure and bail the well down to a greater depth during testing. With the cement slurry in the annulus lifted high up, a better integrity of the casing string over its greater length is secured in the presence of corrosive stratal waters. Lifting the plugging mixture in the annulus high up (to wellhead) is preferable when there is a substantial difference in temperature between the top and bottom sections of the well.

There exist several methods with the help of which the plugging mixture can be brought up to a considerable height in the annular space, *viz.*:

(1) by applying a two-stage method of cementing with some discontinuity of the cement ring along its height;

(2) by employing a plugging mixture of a lower density, i. e., through reducing the difference between the densities of the cement slurry and the drilling mud, with simultaneous adjustment of other properties of the plugging mixture.

Lightened plugging mixtures have gained wide popularity in the oil-producing districts of the Soviet Union and other countries. The most well known additives used in reducing the density of cement slurries include bentonite, perlite, pozzolan materials, diatomaceous earth, and gilsonite. In using most of the "lightening" agents, the reduction of the density of a cement slurry implies retention by the additives of an excess amount of water, i. e., raising the water-cement ratio, or introduction of air together with the aggregate. In all such cases, the addition of "lightening" agents tends to decrease the mechanical strength of the resulting stone.

It is bentonite that has gained the most widespread application as an agent reducing the density of slurries. It allows the water-cement ratio to be greatly raised and in this way enables one to reduce the density of the slurry, without making the latter suffer any noticeable density stratification.

Perlite also helps retain a large amount of water. As a rule, it is introduced into cement together with a small quantity of bentonite to avoid flotation of the perlite particles and to obtain a more homogeneous slurry. Slurries with an addition of perlite have, however, a tangible disadvantage: perlite disintegrates under the effect of pressure, so that the density of the slurries grows higher, which may lead to complications.

An addition of pozzolan materials brings down the density of cement slurries but insignificantly, down to 1.6 g/cm^3.

The use of diatomaceous earth for cementing wells in the USA facilitates introduction of a large amount of water into cement slurries. Water lowers the strength of cement stone, but, after revision of requirements for the strength of the stone, the addition of diatomaceous earth has found wide application in practice. Specimens prepared with diatomaceous earth have shown an elevated resistance to attack by corrosive media.

Gilsonite is a solid hydrocarbon, a variety of natural asphalt, and mineralogically it is classed with asphalts. It has a granular structure and a low density (1.07 g/cm^3). It is virtually impervious and chemically neutral in cement slurries. Its presence in the slurries does not affect the action of diverse setting accelerators. An addition of gilsonite to cement in the ratio 1 : 4 to 1 : 2 suffices to effectively control the loss of cement slurry circulation. Gilsonite demands introduction of a lesser amount of water than do other "lightening" agents, and because of this, the stone has a greater strength. But in a low-viscosity cement slurry, gilsonite, due to its low density, is apt to float up, and this makes it necessary to exercise special caution and introduce some amount of bentonite into the slurry.

In cementing oil and gas wells, three methods of obtaining light-weight cement slurries are employed.

1. Intermingling of cement powder with an additive having a lower density and requiring no water. An example of such an additive is gilsonite. The range of density changes is not very wide.

2. Commingling of cement powder with an additive demanding water. As a result of introducing an excess amount of water (till a slurry of desirable mobility is obtained) the density of the slurry can be changed quite significantly. Among examples of such an additive are clay, opoka, etc.

3. Commingling of cement powder with additives containing air (expanded clay, ashes, perlite, for instance). The density of the slurry can be made low, however, as follows from the data listed in Table 31, it sharply increases when the slurry is subjected to the action of hydrostatic pressure.

Analysis of the figures in Table 31 shows that hydrostatic pressure tends to significantly raise the density of cement-perlite slurries. The magnitude of changes in the density of the slurries under different conditions cannot be predetermined so far. With mounting pressure, the volume of cement-perlite slurries diminishes by 15 to 30 %, and this should be taken account of in practical work.

As regards their preparation and use, most simple are cement-bentonite slurries.

Table 31

Density of Cement-Perlite Slurries as a Function of Hydrostatic Pressure

Slurry composition, kg			Pressure, MPa	Initial density of slurry, ρ_0, g/cm^3	Density of slurry under hydrostatic pressure, ρ_p, g/cm^3	$\rho_p-\rho_0$, g/cm^3
cement	perlite	water				
100	25	100	10	1.31	1.52	0.21
			30		1.52	0.21
			50		1.56	0.25
100	33	145	30	1.19	1.42	0.23
			50		1.44	0.25
100	50	210	30	1.05	1.35	0.30

Lightened cement slurries can be obtained in three different ways.

The binding material is mixed with a clay (bentonite) mud, or else a blend of a binder and bentonite is mixed with water. In principle, the use of both compositions is possible, but it is simpler to make a slurry with a specially prepared mud, though the preparation of such a special mud presents certain difficulties. It often proves expedient to utilize the drilling mud available at the oil derrick, but this must then be tested in a tentative slurry.

The second method, based on mixing with water of thoroughly mingled cement and clay, is used with success in many oil and gas districts of the Soviet Union. When dry materials are mixed with water, the mixture may get lumpy and the density of the slurry may be greater than in the first case. Nonetheless, a careful control and preparation of the mixture enable these shortcomings to be eliminated.

Figure 84 presents a diagram illustrating the preparation of lightened plugging mixtures with plugging portland cement and powdered bentonitic clay as their main components.

This method of obtaining lightened cement-bentonite slurries may result in the development of slurry blocks of high or low viscosity (because of nonuniform mingling of clay and cement).

By employing the first procedure it is possible to produce lighter slurries, but, in spite of the listed advantages, their use is recommended only in cases of uncomplicated cementing jobs, and this is because of the following: (a) in conditions prevailing at an oil derrick, it is impossible to rigidly adhere to the slurry formulation designed at the laboratory; both the rheological properties of the slurries and the physical and mechanical characteristics of stone, being directly dependent upon the degree and quality of the drilling mud treatment, will change sharply, and this may have quite unexpected results; (b) in this particular case, the physical and mechanical properties of cement-bentonite stone are poorer than those obtained by the second method with which, furthermore, the expansion of clay unbound with water in the annular space of the well is ensured.

A third method of preparing light-weight plugging mixtures is also possible, *viz.*, ready-made bentonite mud and cement slurry are delivered by pressure lines to the cementing head where they are mixed in the 1БМ-700 Model manifold unit and pumped into the well. The procedure is simple, but requires considerable preparatory work.

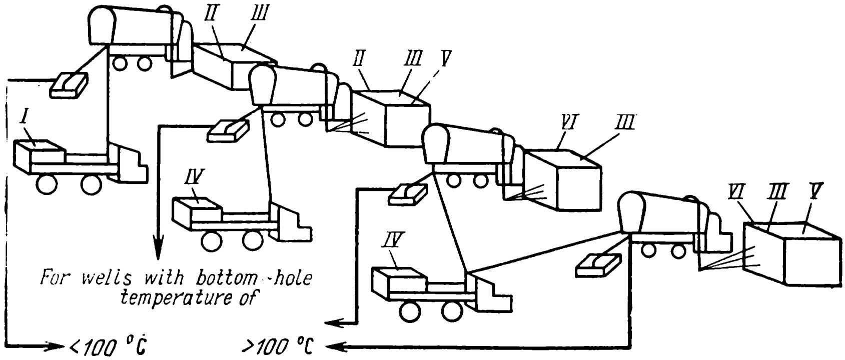

Fig. 84. Process flow diagram for preparation of lightened plugging mixtures
I — water and viscosity reducing agents; *II* — portland cement; *III* — bentonitic clay; *IV* — water, cement setting retarders, and viscosity reducing agents; *V* — powdered opoka; *VI* — slag cement

Depending upon the properties of the cement and bentonite used, the slurries can differ somewhat from one another, but the results of physical and mechanical tests are nearly the same for all the three methods of making the slurries.

Normally, the setting of the above slurries takes a longer time, but at 75 °C they set comparatively quickly. With high proportions of the drilling mud and relatively low temperatures, an addition of accelerators is advisable. With the ambient temperature and pressure rising up to 120 °C and 40 MPa, respectively, the slurries set so quickly that they become practically unfit for use without retarders. The treatment of light-weight slurries with retarders and accelerators does not differ in any way from the analogous processing of ordinary slurries. It is more practical to dissolve the retarding agent in water prior to mixing it with bentonite. As the content of bentonitic clay in the slurry is increased, the density of the latter is observed to lower progressively. The introduction of coarse or medium sand always increases the density of the slurries, but when the temperature in the well exceeds 100 °C, sand must be added to the slurry for the reasons mentioned earlier in the text. In this case, it is advisable to use fine or ground sand, for such a sand requires an additional amount of water for wetting and thus compensates somewhat for the increase in the density of the slurry.

Increasing the content of bentonite in the slurry in all cases reduces its density and lengthens its setting time and also reduces the strength of the resulting stone. With any proportion of bentonite, the strength of cement stone specimens hardening in water at 75 °C is observed to improve with age. An excess of water in the slurry increases the permeability of the stone and reduces its corrosion resistance.

Cement-bentonite slurries are recommended for use not only in difficult conditions of drilling. They may be successfully employed in wells drilled with the use of drilling muds of normal density.

Cement-bentonite slurries possess a well-marked structural viscosity whose minimal value lies in the range 0.12 to 0.2 Pa s. With rising temperature, a tendency towards an increase of viscosity is observed. This fact should not be disregarded in cementing wells, for in some cases the thickening of slurries leads to a sharp rise of pressure

The need for bringing down the viscosity of cement-bentonite slurries is quite obvious. This can be achieved by raising the water-cement ratio of the slurries, or by their treatment with chemical agents. The second procedure is most acceptable, since its use makes it possible to obtain mobile mixtures with their structural-mechanical properties remaining practically stable in the course of cementing, without any impairment of the physical and mechanical properties of the stone and without any abrupt variations in the density of the mixtures.

Cement-bentonite slurries are thinned out with sulphite-alcohol liquor.

At a temperature of 22 °C, the strength of two-day old cement-bentonite specimens is low; with the slurry composition ranging from 4 : 1 to 3 : 2, the compressive strength of the specimens varies from 1.0 to 0.3 MPa. The compressive strength values for 28-day old specimens of the same compositions are 9.0 and 3.5 MPa, respectively.

Increasing the ambient temperature to 75 °C improves the strength of cement-bentonite stone. Thus, the compressive strength values for the 2- and 28-day old specimens of the above compositions are in this case 9.0 and 5.0 MPa and 10.0 and 7.5 MPa, respectively.

Introduction of opoka into the slurries helps improve the quality of cement-bentonite stone in almost all cases. The permeability of cement-bentonite stone, as determined by the adopted method (i. e., drying of test pieces at 105 °C till steady mass) is low: after hardening at 75 °C during 7 days and more, it diminishes with the age of the stone. At 22 °C, because of their low strength,

Basic Characteristics of Mixtures and Slurries with Different Proportions of Portland Cement and Bentonitic Clay Powder

Composition of mixture, kg		Cement/clay ratio, by mass	Density, g/cm^3	Water/mixture ratio, by mass	Standard fluidity cone test, cm	Approx. volume of slurry from 1 t of mixture, m^3	Approx. volume of 1 t of dry mixture, m^3	Equivalent quantity of mixture in lieu of 1 t of cement to obtain an equal volume of slurry
cement	clay							
1000	0.00	1 : 0	1.85	0.50	22	0.82	0.32	1.00
800	200	4 : 1	1.56	0.84	18	0.17	0.33	0.70
750	250	3 : 1	1.50	0.93	18	1.27	0.34	0.65
670	330	2 : 1	1.46	1.05	18	1.39	0.34	0.59
600	400	3 : 2	1.45	1.05	17	1.40	0.35	0.59
500	500	1 : 1	1.38	1.30	18	1.65	0.35	0.50

the specimens disintegrate while being dried. After air seasoning for 7 days, the permeability of the specimens is insignificant. Increasing the ambient temperature and pressure up to 100-110 °C and 40 MPa, respectively, causes a sharp decrease in the permeability of cement-bentonite stone after two days of hardening, which allows the slurries to be recommended for use at temperatures up to 100-110 °C.

Table 32 lists basic characteristics of mixtures and slurries with varying proportions of portland cement and bentonitic clay, recommended for use in the field. In all cases, portland cement and powdered clay are preliminarily mingled together and then mixed with water; the mixture is stirred for 3 minutes. From Table 32 it is evident that with an increased proportion of powdered clay the yield of the slurry becomes much higher.

10.1. Lightened Plugging Mixtures with Finely Ground Silica Additives

With temperatures exceeding 100 °C and high pressures, it is expedient to introduce into cement-bentonite slurries ground silica additives which, combining with calcium hydroxide that is being released, form low-basicity hydrosilicates which are stable under given conditions.

The density of cement slurries can also be reduced by increasing their water-cement ratio, but a finely ground silica additive can play the part of a "lightening agent". The density of the slurry can in this case be brought down to 1.5 g/cm^3.

Among silica additives employed to cut down the density of plugging mixtures are diatomaceous earth (diatomite), pumice, opoka, and tripoli.

The lightened plugging mixtures carrying additives of the above-mentioned silica materials have an advantage of producing a stone with better physical and mechanical properties when it is solidifying in conditions of high temperatures and pressures.

Their shortcomings are a greater water loss and a tendency toward density stratification.

To lengthen the setting and thickening times of the slurries at high temperatures and pressures, one should make use of retarding agents. It should then be borne in mind that some amount of such an agent is adsorbed on the surface of clay or silica additive and, therefore, a larger quantity of the retarder relative to cement should be taken. In this particular case, cement-bentonite slurries require an addition of a relatively greater amount of the retarding agent.

Developed in this country is a plugging cement containing tripoli. The mixture contains 47 % clinker, 50 % tripoli, and 3 % gypsum and enables one to prepare a slurry having a density of 1.5 g/cm^3 at a water-cement ratio of 0.9. The initial viscosity of the slurry is 0.6 Pa s; the slurry has a somewhat lowered water loss and is relatively easy to treat with the CMC setting retarder.

10.2. Lightened Slag Slurries

Since at high temperatures and pressures the physical and mechanical properties of plugging portland cement stone are liable to deteriorate, it is practicable to utilize slags for the preparation of lightened cement slurries.

Extensive use is made of lightened slag slurries with bentonitic clay and also with bentonitic clay and opoka. Slags can serve as a principal constituent in making lightened plugging mixtures, both with finely ground siliceous and argilaceous materials.

Most widely used are lightened slag slurries with bentonitic clay and also those with bentonitic clay and opoka.

Figure 85 illustrates changes in the density of a slag-bentonite slurry with growing proportions of bentonite and water. Different types of slag respond dissimilarly to the introduction of bentonitic clay, but under all conditions the setting rates become progressively slower (Fig. 86). Already upon introduction of 30 to 50 % of bentonitic clay, some kinds of slag are apt to lengthen the initial set time to such an extent that the mixtures become practically little fit to be used in the test conditions. For this reason, they ought either to be used at higher temperatures or be treated with activators. Introduction of 43 % of clay (by mass of stale slag), for instance, tends to retard the initial set of the slag-clay slurry at 130 °C and a pressure of 40 MPa roughly from 20 minutes to 1 hour 20 minutes. Introduction of 100 % clay makes it possible to retard the initial set of the slurry for as long as 6 hours 30 minutes. Raising the temperature up to 150 °C helps reduce the setting time, but its absolute value (4 hours

20 minutes) remains (with the ratio 1 : 1) long enough to warrant cementing.

The use of stale slag enables one to obtain slag-bentonite slurries with a still longer setting time. It can serve as a principal ingredient for preparing slurries with satisfactory setting rates

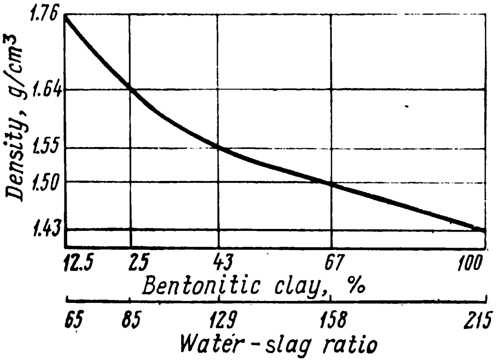

Fig. 85. Effect of bentonite and water content on the density of slag-bentonite slurry

at higher temperatures. Introduction of opoka together with clay helps improve the structure of slag-bentonite stone, raise its strength, and lower its permeability.

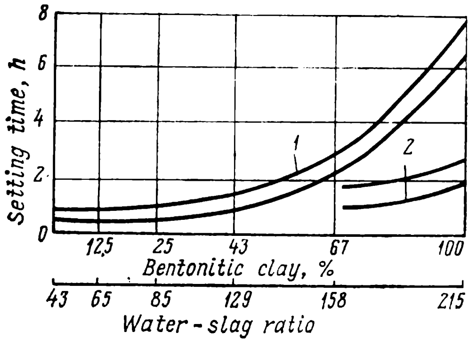

Fig. 86. Effect of bentonite and water content on the setting time of slag-bentonite slurry

1 — t = 130°C, p = 40 MPa (freshly ground slag); 2 — t = 170°C, p = 40 MPa (stale slag)

Addition of large amounts of bentonitic clay to slag cement contributes, contrary to the prevailing opinion, to a higher mechanical strength of slag-clay stone, provided the proportion of clay does not exceed approximately 50 to 60 % of the mass of slag (Fig. 87). The mechanical strength of the specimens is seen to go up with the age of the stone (at 130 °C). At 170 °C and a pressure of 40 MPa, the strength of pure slag stone specimens is somewhat greater than that of specimens containing clay. Nonetheless, in this case the absolute values of stone strength are higher than at 130 °C. Addition of opoka also contributes to the growing strength of the stone.

As the clay content in a specimen grows higher, its porosity increases, whereas its density and unit bulk mass go down.

Lightened slurries with slag as their principal component may be prepared by mixing slag with a bentonite slurry prepared earlier. There have also been developed some other lightened plugging mixtures with portland cement, slag and lime as their main constituents. Various Soviet research bodies have evolved a lime-silica cement composition that can serve as a basis for obtaining slurries with a density of 1.25 to 1.27 g/cm^3.

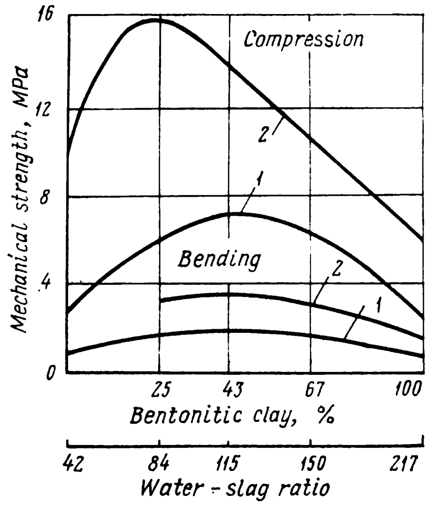

Fig. 87. Effect of bentonite content on the mechanical strength of two-day old slag-bentonite specimens

1 — t = 130°C, p = 40 MPa; *2* — t = 170°C, p = = 40 MPa

10.3. Weighted Cement Slurries

Weighted plugging cements are used in cementing oil and gas wells. Adding weight to cement slurries is advisable only in cases of extreme necessity, i. e., in cementing wells that are drilled with the use of mud fluids whose density is around 1.9 g/cm^3 and even more.

An extra weight can be added to cement slurries in two ways, *viz.*, by manufacturing iron-ore cements and through introduction into cements of agents adding weight to them. The first procedure provides for obtaining cement slurries of high density, while the second, i. e., commingling of cements with powdered weighting materials, makes it possible to adjust density over wide limits, depending upon the quantity, density, granulometric composition, and moisture-retention capacity of the weighting agent. To keep down the quantity of water so as to increase density, it is advisable to introduce into the slurry plasticizing agents, the most effective of which are spent sulphite-alcohol liquor, sulphite-cellulose extract, and others.

Table 33 lists some physical properties of weighting agents.

It is seen from Table 33 that the density of weighting agents is high enough, reaching 7.69 g/cm^3 in the case of ferrophosphorus.

Table 33

Some Physical Properties of Weighting Agents

Physical properties of weighting agents	Pyrites cinder	Hematite	Magnetite	Ferrosilicon	Specular iron	Ferrophosphorus
Density, g/cm^3	4.29	4.35	4.52	6.52	6.55	7.69
Volume (unit) weight, g/cm^3:						
loose	1.87	1.89	2.39	2.13	2.14	3.64
consolidated	2.26	2.32	2.81	3.09	2.93	4.76
Maximum grain size, mm	0.15	0.20	0.30	0.15	0.15	0.20

The presence of admixtures in hematite and, especially, in pyrites cinder demands introduction of an excess amount of water into the slurries, which reduces the density of weighted cement slurries. The density of a slurry carrying one and the same weighting material can be increased by grinding the latter coarser, for in this case less water is needed to wet it. Quite often, a coarse-grained weighting agent of a lower density provides for obtaining a slurry of a higher density than this can be achieved by using a finely ground weighting material of a higher density. The first three weighting agents are quite frequently used in the field. Their relative hardness is pretty high, the Brinell ball hardness of specular iron being 350 and that of ferrosilicon, 220. Nevertheless, this circumstance cannot present a serious obstacle to their utilization as weighting agents, for their cost is low and they are highly effective. The same purpose is also served by barite.

Table 34 lists some data on the influence of weighting materials on the properties of cement slurries that underwent maturing at 90 °C for a period of two days. Addition of hematite to cement helps make slurries of a fairly high density and it is possible to obtain a slurry with a density of 2.2 g/cm^3 at a ratio of cement to hematite of 1 : 2. With increasing quantity of hematite in the slurry, the mechanical strength of specimens goes down.

Addition of SAL helps slow down the setting rate, decrease the consumption of water and thereby increase the density and the strength of specimens. Much weight is added to cement slurries by using magnetite (magnetite sand). The latter requires less water than does hematite.

Because of their relatively low density and high degree of contamination with rock which requires an excess amount of water, pyrites cinder cannot be of any significant help in augmenting the density of the slurry. Addition of ferrosilicon and specular iron does not produce any anticipated effect, in spite of their high density, in view of the high water-requirement of the powder.

Table 34

Changes in the Properties of Cement Slurries Hardening at 90 °C, Following Introduction of Various Weighting Agents

Slurry composition by mass, %			Density, g/cm^3	Setting time, h. min		Ultimate strength in 2 days, kg/cm^2		Porosity, %
cement	weighting agent	water		initial set	final set	in bending	in compression	
100	0	40	1.90	1-26	1-46	68	216	37.9
100	0	50	1.83	1-50	2-40	47	93	46.5
Hematite								
100	50	61	1.98	1-31	2-26	36	63	44.2
100	100	74	2.09	1-37	2-40	28	38	44.8
100	150	88	2.17	1-39	2-41	20	27	44.9
100	200	100	2.20	1-41	2-43	14	23	45.0
Magnetite								
100	50	53	2.08	1-96	2-13	43	77	43.2
100	100	55	2.32	1-33	2-29	40	73	39.3
100	150	58	2.48	1-25	2-15	39	71	36.0
100	200	61	2.60	1-22	2-09	35	65	35.4
Ferrosilicon								
100	70	62	2.18	—	6-33	28	53	44.9
100	100	70	2.29	6-10	7-28	26	52	46.5
100	150	81	2.42	6-26	8-18	25	39	47.9
100	200	88	2.56	6-51	8-21	22	38	49.5
Specular Iron								
100	70	61	2.19	6-09	6-53	32	73	44.3
100	100	69	2.29	7-01	8-06	29	71	45.7
100	150	76	2.43	7-14	8-13	—	69	45.9
100	200	79	2.62	7-25	8-37	28	60	46.5
Ferrophosphorus								
100	50	56	2.11	3-00	3-50	38	59	42.0
100	100	59	2.45	4-23	5-57	34	—	45.0
100	150	65	2.69	4-33	6-46	32	47	47.5
100	200	67	2.89	4-31	6-49	31	46	47.7

The application of pure weighting agents of high density at a definite (optimal) granulometric composition permits obtaining plugging mixtures of high density.

The quality of a material as a weighting agent for cement slurries may be appraised in terms of its specific surface, density, purity, water-requirement and granulometric composition. To obtain cement slurries of high density, it does not suffice to have a weighting material of high density; the latter must have a definitely set specific surface and be devoid of hygroscopic additives.

And, conversely, by using some material of a comparatively low density (2.5 to 3.0 g/cm^3) but a small specific surface, it becomes possible in individual cases to add much to the weight of cement slurries. Therefore, it would be wrong to believe that the weighting agent has to be always milled to size as small as that of cement flour. This would be unjustified, technically disadvantageous and uneconomic.

The specific surface of the weighting agent should be chosen (when technically possible) in each concrete case by taking into consideration the density of the slurry and retaining or improving its other characteristics. Iron oxides or other heavy materials whose granulometric composition is limited to the screen size of 0.60 to 0.15 mm, or if the fraction in question is even more narrow (0.6 to 0.4 mm), may be considered high-grade weighting agents for raising the density of cement slurries. The upper limit for the grain size of the weighting agent must be selected so as to provide for the absence of sedimentation and keeping in good order the equipment during the transport of the cement slurry into the annular space of the well. It is but natural that the notion of the weighting agent fineness implies different meaning when applied to a mud fluid or a cement slurry.

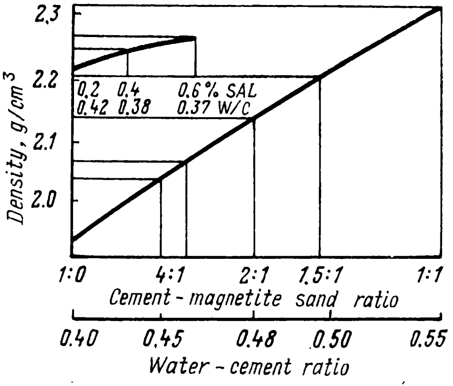

Fig. 88. Effect of magnetite sand content on the density of weighted portland cement slurries

By introducing magnetite sand into portland cements it is possible to obtain comparatively heavy cement slurries (Fig. 88). Thus, 50 to 100 % of magnetite sand in portland cement increases the density of the slurries up to 2.14-2.33 g/cm^3. The greatest proportions (up to 0.4-0.5 %) of spent sulphite alcohol liquor help to thin out the slurries and thus reduce the amount of water in them and in this way to add to their density. With a portland cement-to-magnetite sand ratio of 1 : 1 and 0.4 to 0.6 % of SAL, a slurry with a density of 2.45 g/cm^3 can be obtained.

When employing quartz sand of natural size as a material whose water-requirement is much interior to that of cement, slurries with a density of 2.05 g/cm^3 can be produced. Addition of SAL in this case tends to increase the density but insignificantly. The technique of composing weighted cement slurries that are to be used in deep, high-temperature wells has its own peculiarities. The retarder is dissolved in an amount of water chosen for mixing. After defining and adjusting the fluidity of the slurry, its density is measured, which can be increased by lowering the water-cement ratio or increasing the proportion of the weighting agent in the mixture.

Cement slurries are weighted by using quartz-magnetite sand. Since the specific surface of this kind of sand is small, its wetting requires insignificant quantity of water and, hence, the density of the cement-sand mixture can be raised quite substantially.

In the USA, it is largely barite ground to different sizes, and titanic iron ore that are employed for adding weight to cement slurries. They help raise the density of the slurries up to 2.10-2.25 g/cm^3. This purpose is also served by the Ottava sand. As a weighting material, ilmenite (magnetite sand) has many advantages over barite. It is possible to obtain through a special fractionation a weighting agent that requires the least amount of water. To the category of highly effective weighting agents may be referred galena and iron arsenite, which accelerate somewhat the setting of the slurries. But they are not used in the field because of their high cost.

10.4. Weighted Slag Slurries

Weighted slag slurries are used at high temperatures and pressures. Weighting agents here are the same materials that serve to raise the density of plugging mixtures based on portland cement.

Slag cement has a lower density in comparison with plugging portland cement, but plugging mixtures with a density of 2.20 to 2.25 g/cm^3 can be prepared by using slag as their basic material.

Introduction of hematite, magnetite, and other weighting agents is helpful in retarding somewhat the setting of slag slurries. Best results have been achieved by employing magnetite (Table 35).

10.5. Aerated Cement Slurries

The pressure on the bottom of wells must be reduced when drilling over depleted deposits and also deposits with a low formation pressure and high permeability of rocks. One of the effective measures for failure-free sinking and cementation of wells is the application of aerated drilling muds and cement slurries.

This purpose may be served by aerated cement and cement-bentonite slurries and also slurries treated with surfactants.

Cement and cement-bentonite slurries can be readily and uniformly aerated during their mechanical agitation. The slurries (especially cement ones), however, are liable to degas relatively quickly.

A sulphonol solution added in an amount of 0.25 to 0.40 % to the mixing water of a cement or cement-bentonite slurry raises

Table 35

Changes in the Density of Slag Slurries Due to Introduction of Various Weighting Agents

Slag-to-weighting agent ratio	Water-slag ratio	Standard fluidity cone test, cm	Density, g/cm^3
1 : 0	0.50	25	1.78
1 : 0	0.40	17	1.90
Magnetite Sand			
3 : 1	0.45	17	2.05
2 : 1	0.45	16	2.13
1 : 1	0.52	16	2.26
Hematite			
3 : 1	0,52	18	2.03
2 : 1	0.57	16	2.08
1 : 1	0.67	16	2.15
Granulated Sand			
3 : 1	0.47	18	1.98
2 : 1	0.45	17	2.06
1 : 1	0.52	16	2.14
Hydraulic Fracturing Sand			
3 : 1	0.45	17	1.98
2 : 1	0.45	16	2.02
1 : 1	0.50	16	2.10
Kuban Sand			
3 : 1	0.47	19	1.95
2 : 1	0.47	18	1.98
1 : 1	0.55	18	2.03

its degree of aeration up to 20-22 and makes the slurry more stable. Naphthenate soap is also used for this purpose.

Once some of the entrapped air has escaped and the slurry hardened, the stone shows adequate strength and reduced permeability.

Plugging mixtures are aerated with the aid of compressors and then immediately pumped into the well by cementing units.

Practical Studies

Determination of the Quantity of the Materials Required to Prepare the Needed Volume of a Plugging Mixture.

The needed density (ρ) of the plugging mixture is fixed to suit the given geological conditions and the ratio k of the mass of the cementing material to that of the aggregate is then chosen.

The amount of cementing material, P_{cm} (cement, slag, gypsum, etc.), aggregate, P_{ag} (sand, opoka, etc.), and mixing fluid, P_{mf}

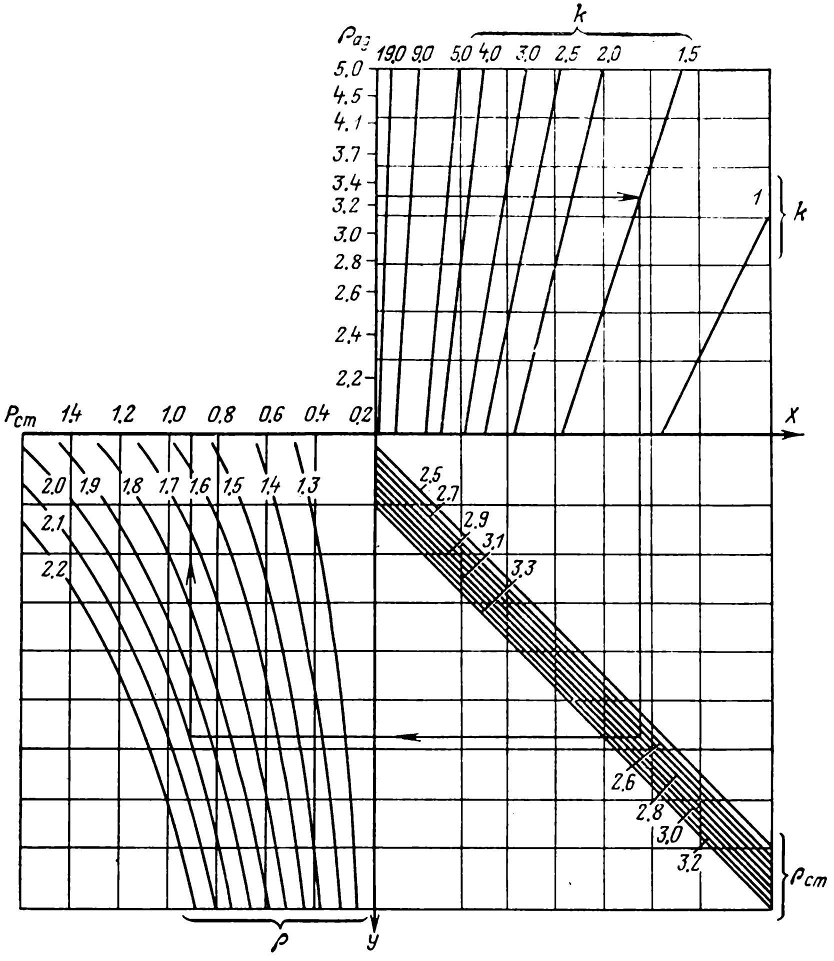

Fig. 89. Nomograph for determining the mass of cementing material required to prepare 1 m^3 of a plugging mixture of specified composition and density

(water, salt-containing solutions, etc.), required to prepare 1 m^3 of the mixture with the specified density is determined from analytical relationships obtained by solving simultaneously the following equations:

$$P_{cm} = (\rho - \rho_{mf})/[(\rho_{cm} - \rho_{mf})/\rho_{cm} + (1/k)(\rho_{ag} - \rho_{mf})/\rho_{ag}] \qquad (46)$$

$$P_{ag} = P_{cm}/k \qquad (47)$$

$$P_{mf} = \rho - (P_{cm} + P_{ag}) \qquad (48)$$

where ρ_{cm}, ρ_{ag}, ρ_{mf}, and ρ are the respective densities of the cementing material (e. g., cement), aggregate (filler), mixing fluid, and plugging mixture, t/m^3.

In most cases, it is water that is used for mixing slurries. In this case, $\rho_{mf} = 1.0$ t/m^3, and so the amount of mixing water required to prepare 1 m^3 of the plugging mixture is

$$P_w = \rho - (P_{cm} + P_{ag}) \tag{48'}$$

Let us reduce the equation (46) to the following, more simple form:

$$P_{cm} = (\rho - 1)/1 - (1/\rho_{cm}) + (1/k)(1 - 1/\rho_{ag}) \tag{49}$$

However, computations by the equation (49) involve some difficulties, and so it is more convenient to use for the purpose the nomograph of Fig. 89.

Example. Find the amount of slag (in tons) required to prepare 1 m^3 of a slag-sand slurry with the density $\rho = 2.0$ t/m^3 at $k = 1.5$ (3 : 2). The starting materials: ferromanganese slag ($\rho_{cm} = 2.8$ t/m^3) and magnetite sand ($\rho_{ag} = 3.3$ t/m^3). The mixing fluid — water.

Solution. Find on the ρ_{ag}-axis the point corresponding to 3.3 t/m^3 and draw from this point a straight line parallel to the X-axis till intersection with the line $k = 1.5$. From the point of intersection drop a perpendicular to intersect the line $\rho_{cm} = 2.8$ t/m^3 and then draw from the intersection point thus obtained a straight line parallel to the X-axis until intersection with the curve $\rho = 2.0$ t/m^2. Finally, erect a perpendicular from the intersection point on the curve to intersect the P_{cm}-axis and find the sought value of P_{cm} : $P_{cm} = 0.91$ t/m^3.

According to the above formulas, the remaining values will be as follows: $P_{ag} = 0.91 : 1.5 = 0.61$ t/m^3 and $P_w = 2 - (0.91 + 0.61) = 0.48$ t/m^3.

Laboratory Work No. 7

I. *Selecting the composition of plugging mixture of low density.*

Prepare a dry mixture of cement and bentonite powder in the ratio 2 : 1 (200 g of cement and 100 g of bentonite powder). Thoroughly mix the components and then pour 200 cm^3 of water into it. After stirring the slurry for 3 to 5 min, determine the following:

(a) fluidity of the slurry (this must be no less than 18 cm in the standard fluidity cone test; should the fluidity of the slurry prove to be inadequate, add more water);

(b) density of the slurry;

(c) setting time of the slurry at 75 °C.

II. *Selecting the composition of a weighted cement slurry.*

Prepare a dry mixture of cement and hematite in the ratio 2 : 1 (300 g of cement and 150 g of hematite). Thoroughly mix the constituents and then add water (approximately 160 cm^3) to the mixture. After stirring the slurry for 3 min, determine the following:

(a) fluidity of the slurry (this must be no less than 18 cm in the standard fluidity cone test);

(b) density of the slurry;

(c) setting time of the slurry at 75 °C.

III. *Selecting the composition of a weighted cement slurry with a specified density.*

First find the required amounts of the constituents by using the nomograph of Fig. 89 and the formula (47). Mix the required amount of cement with that of the available weighting agent of specified density. Add water in an amount approximately equal to one-half the required mass of cement plus 2 to 3 % for wetting the weighting agent. Should the amount of water prove too large and the density of the slurry insufficient, add 0.2 to 0.3 % SAL, while simultaneously reducing the amount of water, and then determine the fluidity of the resulting slurry. The slurry is considered ready if it has the specified density at a fluidity of no less than 18 cm.

Chapter 11

Cement Slurries Prepared with Concentrated Saline Solution (Brines)

A substantial part of oil and gas fields is confined to subsalt and intersalt deposits. Drilling and casing of deep wells in saline deposits entails considerable difficulties even to this day. Among the most frequently encountered minerals are halite (NaCl), sylvine (KCl), bishofite ($MgCl_2 \cdot 6H_2O$) and carnallite ($KCl \cdot MgCl_2 \cdot 6H_2O$). In drilling, saline deposits, which have varying physicochemical and mechanical properties, become subject to intensive caving. Salts contained in solutions in different concentrations affect dissimilarly the properties of plugging mixtures. For instance, the saturation of a drilling fluid with magnesium chloride greatly reduces the caving rate.

One of the principal causes responsible for substandard (poor) cementing of wells sunk in saline deposits is considered to be the dissolution of salt in the cement slurry, which results in the formation of a gap filled with a saline solution between the wall of the well (made up of salts) and the plugging mixture. In the contact area, the plugging mixture fails to set since it is highly diluted with the saline solution. Plugging mixtures are sometimes specially saturated with salts in order to prevent (or keep down) the dissolution of deposit salts. Such plugging mixtures are not susceptible to any significant changes in their properties subsequent to the ingress of salts into them. These changes lead either to acceleration or slowing down of the setting and thickening of the plugging mixtures, depending upon the nature and concentration of the salts. With salts gaining access into slurries, their properties change also in the presence of diverse aggregates. Most of the plugging mixtures saturated with salt are compatible with commonly employed light and heavy additives.

An important practical advantage of plugging mixtures carrying NaCl is the improvement of their rheological characteristics. Under certain conditions, additions of salt to most plugging mixtures in amounts necessary to accomplish saturation of the mixing water tend to appreciably lower their consistency and critical pumping rate, which largely determines the extent of displacement of the mud fluid. This effect is intensified when adding salt to plugging mixtures containing bentonite. The treatment of plugging mixtures with salts changes quite significantly the behaviour of the plugging stone in the aggressive media of mineralized stratal waters.

11.1. Dissolution of Saliferous Rocks in Plugging Mixtures

Listed below are some data on the solubility of salts in water.

Salt	NaCl	$MgCl_2$	$CaCl_2$	$MgSO_4$	Na_2SO_4	Na_2CO
Solubility:						
g/l	360	588.1	731.9	354.3	168.3	193.9
%	26.4	36.0	42.3	26.2	14.1	16.2

The solubility of salts rises with temperature. The solubility of salt mixtures of dissimilar composition differs from that given above. Anhydrites dissolve in water poorly — up to 2 %. The duration of solvent action of cement slurry on the walls of a well made up of salts is much shorter than that of water.

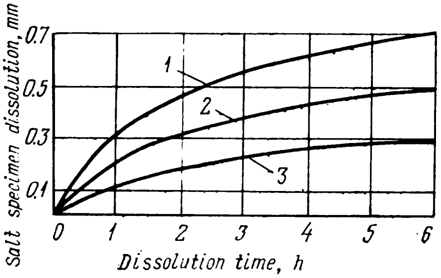

Fig. 90. Dissolution of salt specimens at the surface of contact with hardening cement slurries at 25 °C

With incomplete saturation of cement slurry filtrate, the well walls dissolve less intensively in their static condition after termination of pumping of the slurry into the annulus. The dissolution continues until the cement slurry layer adjoining the saline walls becomes saturated.

The most complete dissolution of salt was observed in cement slurries carrying no salt additions, while with an increased content of salt, the dissolution becomes less intensive.

Figure 90 illustrates the nature of dissolution of salt (NaCl) specimens at the contact between the specimens and hardening cement slurry at 25 °C. The graphs are plotted for slurries without any salt (curve *1*), with 10 % (curve *2*) and 25 % of salt (curve *3*), introduced into the mixing water prior to the preparation of the slurry.

11.2. Preparation of Salinized (Brine) Plugging Mixtures

Analysis of causes leading to complications and of technological features related to natural and artificial mineralization of plugging mixtures and also numerous research works have made it possible to formulate the following conditions for successful completion of cementing jobs and for improving the sealing of beds in saliferous deposits:

(1) application of salt-containing plugging mixtures with necessary properties;

(2) choice of optimal conditions for displacement of the drilling mud from the well;

(3) prevention of intermingling of the drilling mud and plugging mixture in the well.

The choice of the plugging mixture composition and the amount of salts to be introduced is determined by the intensity of the saline deposit dissolution and also by temperature and pressure. Of importance are also the permeability and endurance of the plugging stone and the zone of contact between the latter and the saline deposits.

The ratio between the plugging mixture components depends upon the specified saturation (supersaturation), the achieved concentration of the salt in the aqueous solution used for mixing, and the required water-cement ratio. The density of the slurry increases in all cases of mixing cement with brine water. Salts can be introduced into the plugging mixtures in one of the following ways.

1. Salts are dissolved in the mixing water in amounts providing saturation of water with respect to the salt. In some instances, use is made of supersaturated solutions, be they artificial or natural (brines). Cement is mixed with the adopted saline solution.
2. Plugging cement is mixed with dry salt (powdered) in a preset quantity, with subsequent mixing of the dry mixture with water.
3. Dry mixture of plugging cement and salt is mixed with water in which a definite quantity of salt has preliminarily been dissolved.

At temperatures close to 22 °C, sodium chloride additives in a proportion of up to 5 % speed up the processes of structurization in cement slurries. Increased amounts of salt in the slurry slow down structurization processes. An accurate NaCl dosage, which begins to slow down the structurization of the slurry, depends on the chemical and mineralogical composition of cement. As is known, the composition of cement always varies somewhat, even for cements coming from one and the same plant. Therefore, it is believed that common salt tends to accelerate the setting of a cement slurry made of portland cement at 22 °C, when the amount of NaCl by mass does not exceed approximately 5 %. An aqueous solution of salt is considered to be saturated when it contains 26.4 % of dissolved NaCl.

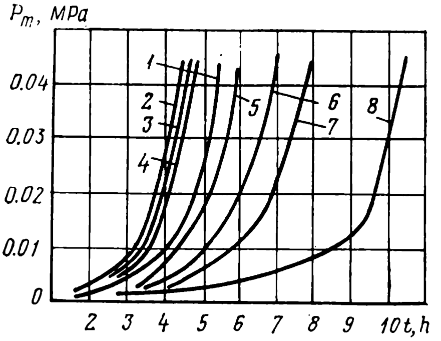

Fig. 91. Effect of salt content on the structurization of cement slurry (t = 22 °C; $W/C = 0.45$)

Mixing fluid: *1* — water; *2* — (2 to 3)-% NaCl solution; *3* — (3 to 5)-% NaCl solution; *4* — 10-% NaCl solution; *5* — 15-% NaCl solution; *6* — 20-% NaCl solution; *7* — 25-% NaCl solution; *8* — 26.4-% NaCl solution

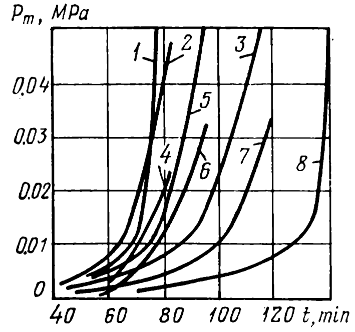

Fig. 92. Effect of salt content on the structurization of cement slurry (t = 80 °C; $W/C = 0.45$)

Mixing fluid: *1* — 26.4-% NaCl solution; *2* — 25-% NaCl solution; *3* — 3-% NaCl solution; *4* — water; *5* — 20-% NaCl solution; *6* — 15-% NaCl solution; *7* — 5-% NaCl solution; *8* — 10-% NaCl solution

Figure 91 shows the effect produced by different concentrations of salt (NaCl) in a cement slurry on its structurization at 22 °C.

With the ambient temperature going up from 22 to 95 °C (Fig. 92), the structurization of plugging mixtures containing any amounts of salt proceeds at a quicker pace. But in the same conditions, NaCl dosages of up to 5 % also tend to slow down the processes of structurization.

Addition of more than 20 % NaCl does not cause any significant acceleration of the structurization processes in cement slurries, i. e., the structurization rate of the slurries with a proportion of salt equalling 20 % and more remains practically the same.

Further increase in the amount of common salt tends to reduce the mechanical strength of cement stone after one or two days' hardening. Determination of the thickening time of NaCl-supersaturated slurries with a consistometer has shown that at temperatures of 130 to 150 °C and pressures of 40 to 70 MPa the tartaric acid retarder used in amounts of 0.5 to 1.0 % is most effective. The structural growth starts quickly and leads to the formation of unpumpable slurries 1.5 to 2 hours after mixing of cement.

With an addition of 5 to 8 % of common salt, the strength of one- and two-day old cement stone specimens maturing at 20 to 50 °C increases.

Addition of carnallite ($KCl \cdot MgCl_2 \cdot 6H_2O$) in an amount of 10 to 30 % enhances the mechanical strength of cement stone. An aqueous solution of carnallite becomes saturated when it contains 30 % of salt. As the concentration of carnallite increases, so does the density of the slurry. A concentration of about 30 % of this salt in a cement slurry leads to the formation of a non-flowing mass.

Structurization of cement slurries even partly salted with carnallite proceeds so intensely that the slurries get thick in a very short time. The effect produced by carnallite gains in intensity as its proportion increases and the ambient temperature gets higher.

Salted plugging mixtures are sometimes difficult to use at elevated temperatures because of their accelerated structurization and loss of mobility. In this case, one should choose adequate structurization (setting) retarders for the plugging mixtures.

It has been found that agents like SAL and CSAL are apt to foam more intensively brine plugging mixtures than slurries prepared with fresh water alone. Salted plugging mixtures may also be treated with CMC and hypan, but these agents tend to lower the fluidity of the slurries. Tartaric acid (TA) and boracic acid (BA) produce no small effect at temperatures over 100 °C and high pressures. The same agents effectively retard the structurization of cement slurries containing elevated proportions of carnallite.

11.3. Effect of Salts on Rheological Properties of Plugging Mixtures

Sodium chloride present in the plugging mixture materially reduces its yield value τ_0 over a long period of time. The plastic viscosity increases somewhat initially.

Introduction of 5 % of sylvine accelerates structurization and improves rheological characteristics of a plugging mixture. An increase in the amount of sylvine till complete saturation of the mixture decreases its yield value, but raises its plastic viscosity.

Bishofite intensifies hydration structurization processes, and so the rheological constants go up. In 30 minutes after mixing, even

15 % of magnesium chloride make the plugging mixture unpumpable. Complete saturation of the mixture results in its instantaneous thickening.

The action of carnallite on plugging mixtures is of a more complex nature. Potassium and magnesium chlorides contained in carnallite produce opposing effects on the structurization of the slurry. Introduction of carnallite that carries more potassium than magnesium chlorides thins out the system, so that its pumping period can be increased; the presence of carnallite containing more magnesium than potassium chlorides accelerates structurization. Antagonism between the potassium and magnesium chloride electrolytes lies in the capacity of K^+ cations to reduce the adsorption activity of Mg^{2+}.

The joint effect of bishofite and carnallite causes the yield value of the slurry to vary over a wide range, from the minimal initial value to the maximum value at the end of measurement. The plastic viscosity changes but slightly.

Dissolution of bishofite in an amount of 15 % in a plugging mixture saturated with halite causes a violent growth of the yield value that already 20 minutes after mixing increases twofold by comparison with the initial figure and very soon reaches its maximum. The rheological characteristics of the same slurry change in a similar manner on introduction of magnesium chloride.

At temperatures up to 90 °C, an effective retarder of structurization of plugging mixtures saturated with bishofite is dextrin. Most of the other additives are not fit to obtain slow-setting plugging mixtures at elevated temperatures.

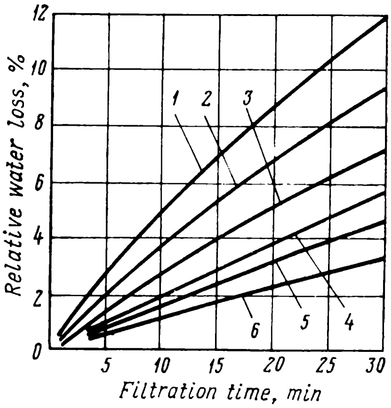

Fig. 93. Effect of salt content on the water loss of cement slurry ($t = 22$ °C; $p = 0.2$ MPa; $W/C = 0.2$)

Mixing fluid: *1* — water; *2* — 5-% NaCl solution; *3* — 10-% NaCl solution; *4* — 15-% NaCl solution; *5* — 20-% NaCl solution; *6* — 26.4-% NaCl solution

11.4. Water Loss of Salinized (Brine) Plugging Mixtures

All other things being equal, different plugging portland cement mixtures containing the same quantities of water and diverse additives (which do not reduce water loss intentionally) feature approximately the same water loss.

An elevated concentration of salts tends to decrease somewhat the water loss of cement slurry. Introduction of combined CSAL and CMC agents into brine cement slurries nearly halves their water loss. Water loss declines more effectively in plugging mixtures salted with carnallite. Water loss is kept down most effectively through preparation of cement-clay (bentonite) mixtures.

Figure 93 shows the dependence of the relative water loss * of a cement slurry during its stirring on the concentration of salt in the mixing water (water-cement ratio 0.5, temperature 22 °C, pressure 0.2 MPa).

11.5. Adhesion of Cement Stone to Salts

The bond between a solidifying cement stone and salts is determined first and foremost by the nature of the cement and the rock, the ambient temperature, pressure, and the condition of the contact surfaces.

As the concentration of salt in the mixing water grows higher, the load required to displace a cement stone specimen relative to a salt core embraced by it is increased. This tendency becomes more and more pronounced as the ambient temperature is increased up to 50 °C. Still higher bond strength values are observed in experiments with expanding cements.

Cement stone adheres better to common salt than carnallite specimens.

11.6. Corrosion of Plugging Cement Stone

An aqueous medium with common salt dissolved therein exerts a damaging effect on the hardening plugging cement stone, irrespective of whether the slurry has been prepared with fresh or salt water.

An addition of 20 to 30 % of quartz sand, opoka, diatomite, or some other material containing a sizable proportion of silica increases the mechanical strength of specimens made of plugging cement mixed with fresh and salt water. At 22 °C, the additives promote the growth of the mechanical strength of cement stone even after one and a half years of its maturing in an aqueous medium having various amounts of common salt.

Active silica in these additives binds free calcium hydroxide and transforms it into calcium hydrosilicate which is virtually insoluble in water and thus raises the strength of the stone.

Cementing of wells in saline deposits with brine cement slurries secures good contact, which is also attributed to some expansion (up to 0.02-0.05 %) of brine cement slurries during their solidification.

At elevated temperatures and pressures, the salt-resistance of cement specimens decreases due to an adverse thermal influence. Mixing of cement with a saturated solution tends to reduce somewhat the stone strength at 110-125 °C.

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* By the relative water loss is meant the ratio between the volume of filtrate and that of mixing fluid.

Carnallite solutions are fairly harmful to portland cement stone. The cement stone specimens maturing in such a solution at 125 °C disintegrate in 1.5 to 2 months. The destruction of portland cement specimens is the greatest in cases where 10 to 20 % of carnallite is added to the mixing water.

At temperatures of 110 to 130 °C and high pressures, it is slag-base specimens with additions of bentonite and quartz sand that undergo slower destruction.

11.7. Features Specific to Cementing of Wells in Permafrost Areas

Depending upon various factors, the depth of rock occurrence with subzero temperatures (—1 to —10 °C) in oil- and gas-bearing areas in permafrost ranges between 100 and 500 m.

Cementing of wells in permafrost regions is a complicated task, and great efforts are required to secure proper sealing of beds.

Experience gained in casing of wells in districts with prevailing perpetual frost demonstrates that the quality of cementing with ordinary plugging cements is not high enough. The cement slurries fail to set for a long time, become congealed, and, once the thermal conditions in the well are disturbed, channels start to form in the annular space. The characteristic feature of many areas is that the temperature of the flushing fluid used in drilling of holes to accommodate the surface casing is usually close to zero. Its increase is followed by defrosting and subsequent instability of the walls in the well. The temperature of the plugging mixture forced into the well is usually 0 to 10 °C.

Cement stone is formed also at temperatures closely approaching zero. Under these conditions, however, the processes of structurization slow down sharply, and much time is needed for the stone to form. Thus, at a temperature above but close to zero, the setting proceeds 4 to 5 times slower than at 22 °C. In this case, the mechanical strength of two-day old stone is but one fifth to one-sixth of that at 22 °C and amounts to 0.2-0.4 MPa in bending.

In the early period of setting. the permeability of plugging stone at low temperatures is pretty high. The mechanical strength of cement stone rises while its permeability diminishes with the passage of time, even when the specimens harden at temperatures close to zero. Once the plugging mixture is defrosted, it gets thick, but hardens only when its temperature is positive. The stone of a cement slurry hardening at temperatures close to zero often carries inclusions of ice whose thawing is followed by the emergence of cracks and sometimes channels. The abundance and the size of the cracks depend upon various factors, among which are the freezing and setting time, thawing temperature, water-cement ratio, etc. Specimens subjected to freezing usually swell and increase in volume.

To eliminate or attenuate the adverse effect of low temperatures on the properties of plugging cements and the quality of well cementing jobs, it is advisable to add setting accelerators to the slurries. The additives or agents intensifying the processes of structurization and hardening should ensure non-freezing of the liquid phase in the cement slurry at the lowest temperatures that may possibly exist at the time of cement setting in the annulus and secure maturing of the stone with its strength growing

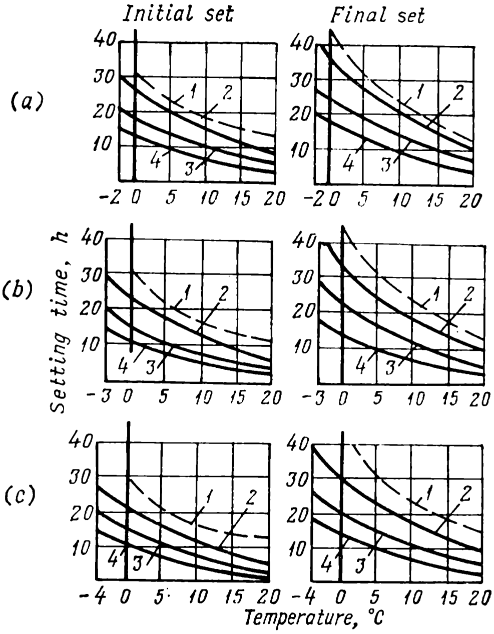


Fig. 94. Effect of temperature on the setting time of cement slurries with additives

Additive content: (*a*) 4%; (*b*) 6%; (*c*) 8%. Additive: *1* — no additive; *2* — NaCl; *3* — $CaCl_2$; *4* — $AlCl_3$

progressively and its permeability diminishing as progressively to certain values over a period of time stipulated by the pertinent standards. Optimal results have been achieved with the use of calcium chloride as an accelerator. At temperatures of 0 to 5 °C, the amount of $CaCl_2$ usually varies from 4 to 10 %. For example, a cement mixed with water carrying 5 % of calcium chloride and subsequently cooled to a temperature of —3 to —5 °C hardens into a stone with satisfactory physical and mechanical properties. One must, however, strictly adhere to the composition formulated at the laboratory.

NaCl is also used as a setting accelerator for plugging mixtures. Its quantity is taken conformable to the slurry composition proportioned at the laboratory, usually at 2 to 3 % of the mass of cement.

Cement slurries are effectively acted upon by aluminum chloride. A 4- to 10- % addition of $AlCl_3$ reduces the setting time of the slurries as much as 5 to 8 times at temperatures of 0 to 5 °C, but this is accompanied by deterioration of their fluidity.

The effect of accelerators depends not only on the type of cement and the ambient temperature, but also on the time and conditions of cement storage. In all cases, accelerators tend to augment the strength of cement stone and reduce its permeability.

Figure 94 illustrates the influence of various accelerators on the setting rates of cement slurries at low temperatures. But the accelerated thickening and setting of the slurries does not fully solve the problem of adequate sealing (separation) of reservoirs in permafrost areas. Recently, new plugging cements have been specially developed for cementing wells in permafrost regions. Serious attention is to be paid to the technology of cementing in conformity with the practice of quick drilling over of the entire permafrost zone. One should watch closely the operations involving any possible disturbance of the heat regime of wells both in drilling and in developing the deposit.

Prior to cementing a well, the mixing water should be warmed up.

Laboratory Work No. 8

Methods of calculation of slurry components and preparation of salt-cement mixtures.

The proportion of components in a plugging mixture is contingent upon the following factors: the specified supersaturation, achieved concentration of salt in the aqueous solution for mixing, and the adopted water-cement ratio.

The procedure and sequence of calculation are as follows. For example, the starting data are: the degree of supersaturation is 35 per cent; the quantity of the aqueous saline solution needed for mixing 1 unit mass of the plugging mixture equals 0.5 volume unit (the true water-cement ratio with due regard for the amount of dissolved salt is 0.45); the density of the fluid prepared for mixing is 1.18 g/cm^3 (with a 23.6- % NaCl concentration).

(1) Determine the amount (in tons) of NaCl needed to obtain a 35- % concentration in the initial 0.45 m^3 of fresh water. With a 35- % concentration, the saline solution consists of 0.65 m^3 (ton) of fresh water and 0.35 ton of salt. Hence, the quantity of sodium chloride (in tons) will be

$$\mathrm{NaCl} = (0.45 \times 0.35)/0.65 = 0.24\ \mathrm{t}$$

(2) Determine the content of NaCl in that amount of the prepared saline solution which includes 0.45 m^3 (ton) of fresh water. With the attained concentration of 23.6 %, the saline solution includes 0.76 ton of fresh water and 0.24 ton of dissolved salt.

Therefore,

$$NaCl = (0.45 \times 0.236)/0.76 = 0.14\ t$$

(3) The quantity of sodium chloride that has to be added to 1 ton of cement is 100 kg (0.24 t — 0.14 t = 0.10 t) and. thus, 50 kg of salt should be added per each sack of cement weighing 50 kg.

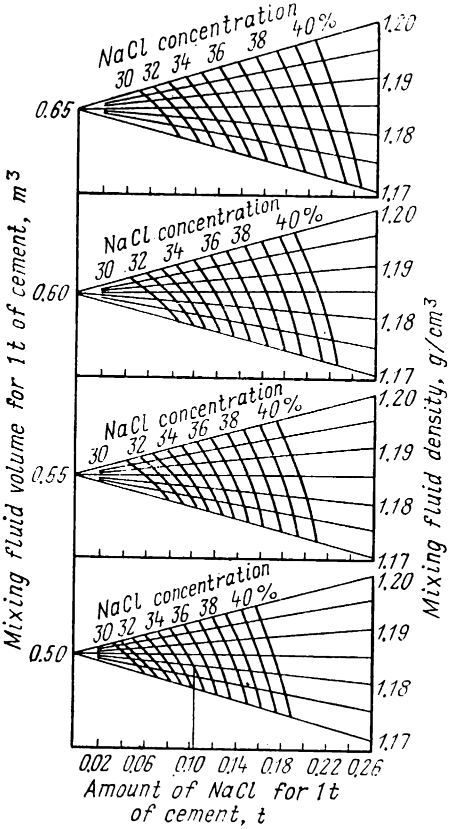


Fig. 95. Nomograph for determining the composition of salt-cement mixtures

In the field practice, there may emerge various combinations of the above factors that determine the proportion of components in a salt-cement mixture. In order to simplify and expedite the obtaining of the necessary data, a nomograph has been compiled (by V. M. Vyazelshchikov). Depending upon the quantity of fluid (Fig. 95) required for mixing of 1 ton of cement, one of the four sections in the nomograph is taken. A perpendicular from the point marking the intersection of the straight line representing the density of the mixing fluid with the concentration curve is dropped onto the horizontal axis on which the amount of sodium chloride to be added to 1 ton of cement is found.

Laboratory Work No. 9

Determining the setting time of a cement slurry.

(1) Pour salt (NaCl) into a vessel containing a measured quantity of water for mixing with cement till the water is saturated at room temperature. Mix cement with the saline solution thus obtained. Determine the setting time of the cement slurry.

(2) Add 5 % by mass of $CaCl_2$ to the mixing water. After its dissolution, mix the whole with the cement. Determine the setting time of the cement slurry.

(3) Make the same experiment, but use NaCl (3 %) and $AlCl_3$ (5 %) in place of $CaCl_2$.

Chapter 12

Plugging Materials for Controlling Loss of Circulation

Drilling mud losses are serious complications, especially if lost circulation is difficult and of frequent occurrence. Loss of circulation may be due to various causes. Sometimes it is caused by voids or cavities exposed by the well, while in other cases, drilling mud is lost into fissures formed as a result of hydraulic fracturing of formations or opening of joints in the rocks under the effect of the high pressure exerted by the fluid column in the well or the elevated pressures attending the movement of the drilling mud or cement slurry through the well. The methods for preventing or controlling lost circulation are as varied as are its causes. Lost circulation control in the event of hydraulic fracturing of formations is frequently effected by reducing the density or flow velocity of the fluids in the well or by chemically treating the fluids, whereas in cases where cavities, fissures or other voids are present in the rocks pierced by the well, drilling mud losses in the course of well sinking are often prevented by employing such plugging materials (referred to as lost circulation, or bridging, materials) as wall of the fissures, set quickly in the voids, thus plugging them up, clog the pores, or thicken while still not very far away from the well, forming a bridging plug that allows the drilling to proceed further. But the best method to control circulation loss is to prevent it.

The key properties of lost circulation (bridging) materials are considered to be the size and shape of their grains and also their rigidity and density. A bridging material is considered fit for an isolation work if its grain size is no less than one-third the maximum size of the pores or fractures to be clogged or bridged. Special materials are used to wall off, or plug in, large-size fractures.

In the practice of controlling lost circulation in drilling, use is made of materials that can be pumped together with the drilling mud and are believed to help prevent or control its loss into the formation. Among such materials are coke, lumps of clay, leather, cellulose flakes, moss, mica, asbestos, expanded clay, feathers, paper (and cement paper sacks), waste products of the textile and rubber industries (selvage, fibres), cotton-seed hulls, wool, felt, perlite and slag grains, sugar cane and corn stalk fibres, soft chips, straw, wheat bran, peas, beans, cotton, and other materials.

The materials for bridging lost circulation (thief) zones are classified into fibrous, lamellated, and granular.

The choice of the type and size of fibre is largely dependent upon the nature of the work to be performed. They are also decisive in determining the quality of the latter.

Fibrous materials render satisfactory results in bridging small crevices in sandy and gravelly rocks with a grain size (diameter) of up to 25 mm.

Lamellated materials are utilized for clogging crevices and pores measuring 2 to 3 mm across.

Granular materials bridge most effectively pores and fissures in beds composed of gravelly rocks with a grain size of 25 to 30 mm. Fragmented rubber, for example, is applied in bridging openings up to 8 mm in diameter, while perlite is used in clogging pores measuring 10 to 12 mm across.

Frequent use is made of mixtures of fibrous and lamellated materials, as well as those of lamellated and granular ones.

Lost circulation control at low pressures is most effectively accomplished by means of a highly colloidal drilling mud carrying additions of fibrous materials (to form a network) and mica.

Lost circulation control consists in transporting the bridging material to the thief zone and forming there a girth capable of withstanding some pressure differential. If pores or fractures are clogged in several operations, each subsequent operation is carried out using bridging materials whose grain size is larger than that of the materials employed in the previous operation.

12.1. Plugging Mixtures for Controlling Loss of Circulation in Drilling

The success of lost circulation control largely depends on the quality of the plugging mixtures used for the purpose.

The plugging mixture intended to control drilling mud losses must possess a number of special features. It must remain fluid while being transported to the thief zone and must set quickly once there, turning into stone in a short time. The stone must not disintegrate under the effect of stratal (brine) waters, temperature and pressure. The components of the plugging mixture should be selected and its properties fixed with due regard for concrete conditions prevailing in a given well.

Many characteristics of plugging mixtures applied to control lost circulation are close to those of the slurries used in casing cementing jobs. The evaluation of both kinds of materials as to their suitability for use in wells is often done along the same lines.

The physical and mechanical properties of the stone of plugging mixtures are not standardized, but the field data and laboratory research give grounds to consider the plugging mixtures hardening into a stone with a compression strength of 3 to 4 MPa and permeability of $(1 \text{ to } 2) \times 10^{-3}$ μ^2 as quite suitable for isolation work.

A major feature of plugging mixtures employed to control lost circulation is the rise in their viscosity on the delivery to the thief zone. But they must preserve mobiltiy during their pumping into the annular space of the well.

In preparing plugging mixtures, use is made of diverse binding bases. Most prevalent are inorganic binders, such as portland cement (plugging, constructional), alumina, gypsum-alumina, and gypsum cements.

Very popular is fibrous cement, especially in bridging relatively large pores and cracks. Its basic component is usually portland cement. Materials of mineral and organic origin (natural and artificial), such as asbestos and leather fibres, ground plastics, nutshells, cotton-seed hulls, etc., are used as a fibrous filler. The fibres of the materials are commonly 8 to 10 mm long. The so-called plastic cements are made by adding powdered rubber to portland cements.

Solar oil-bentonite mixtures are also used to control lost circulation. They rapidly gain in structural strength and thus help achieve effective sealing of the intake bed. But the solar oil-bentonite mixtures have inherent disadvantages, namely, they are sensitive to water inclusions in their composition and exhibit a low mechanical strength.

12.2. Quick-Setting Mixtures

Quick-setting plugging mixtures are very often used to control lost circulation in situations where temperatures do not exceed 50 to 70 °C.

In such conditions, ordinary plugging mixtures and constructional mortars have too long a setting time, and so, having enough time to move far away from the well, cannot ensure proper bridging of the lost-circulation formation. Quick-setting mixtures, having entered fractures and caverns, are diluted to a lesser extent with stratal waters or drilling mud and intensively thicken and harden into stone. The time period marking their transformation into stone is much shorter than in the case of ordinary cement slurries, and so is waiting on cement.

Quick-setting plugging mixtures are usually prepared on the basis of plugging cements, with some structurization (setting) accelerator introduced into the mixing water. Most reagents or procedures speeding up the setting of plugging mixtures tend to increase the mechanical strength of cement stone at an early stage of hardening.

In the practice of controlling lost circulation, it is calcium chloride, $CaCl_2$, that is more often used. Introduction into cement slurries of 2 to 3 % of $CaCl_2$ together with the mixing water shortens the initial set period of the slurries to 30 to 60 minutes and steeply increases the initial mechanical strength of cement stone. To improve the quality of isolating work, a solution of calcium chloride that cleans the walls of the fractures and channels subject to plugging is pumped into the well before any cement slurry is introduced into it.

Common salt (NaCl) in an amount of 3 to 5 % is also used as a setting accelerator for plugging mixtures. When its percentage is increased further, NaCl acts as a retarder.

The same purpose is served by gypsum, which is used either as an additive to plugging cements or independently, where the temperatures in the lost circulation zone do not exceed 50 to 60 °C. In a situation of this kind it is sometimes necessary to use also structurization retarders to provide for the needed setting time of the mixture. This task is fulfilled by the same agents as are used in slowing down the setting of cement slurries in the primary cementing.

Gypsum is habitually added to cement and the two are thoroughly mixed. When mixed with water, the cement-gypsum mixture sets and gains in strength very rapidly, so that 1 to 2 hours after mixing with water, the compressive strength of the resulting stone reaches 10 MPa and more. While hardening, the mixture expands somewhat.

Considerable quantities of water glass contribute to a highly intensive structurization of cement slurry, but its use may complicate the surface equipment layout.

The same purpose is also served by soda ash (in a small concentration), which should be introduced into the plugging mixture (at the time of mixing cement with water) in an amount that must strictly conform to that fixed by the laboratory.

Various kinds of resin are introduced into cements to form quick-setting mixtures. Such systems have definite advantages, but in every case, all plugging mixtures should be subjected to a laboratory check under conditions closely approaching those prevailing in the well.

Soviet research bodies have developed some lightened clay-cement mixtures containing aggregates that are intended for sealing lost-circulation formations in deep wells. The composition of the mixtures includes: mud powder — 50 to 70 %, plugging powder — 20 to 45 %, inert aggregate (expanded clay, quartz sand with particles up to 5 mm across) — 5 %. The density of such mixtures is 1.25 to 1.45 g/cm^3.

It is expedient that lightened cement-bentonite mixtures with inert aggregates be utilized in controlling complete loss of circulation in fissured and cavernous rocks of wells at depths over 3000 m and elevated temperatures in the well bore (70 to 95 °C).

12.3. Gel-Cements

Gel-cements are paste-like slurries consisting of cement and clay (bentonite). They are often prepared by mixing cement with clay drilling mud or water containing some amount of bentonite.

The resulting slurry has a reduced density, is characterized by a sound structure, and hardens into stone. In preparing such slurries, fresh water is sometimes treated with sodium carbonate or caustic soda. Then bentonite is added to water in an amount of 25 to 30 kg/m^3.

To lower the pressure on the formations, plugging mixtures of low density are used in controlling lost circulation. Among these are cement-bentonite and cement-perlite mixtures, mixtures carrying additions of pozzolan, opoka, diatomite, tripoli, pumice, etc. In other countries, use is also made of gilsonite, perlite, and diatomaceous earth.

12.4. Features Specific to the Setting of Quick-Taking Mixtures. Selection of Mixtures for Concrete Conditions

The technology of controlling lost circulation envisages changes in the state of the plugging mixtures, from liquid, fluid (at the time of pumping) to viscous-plastic (when they gain access into crevices and channels), followed by their transformation into a lithoidal body as the work in the well progresses.

The most important properties of quick-setting plugging mixtures are their structural and mechanical characteristics. Their key characteristic, the mechanical strength of their structure, may give an idea as to their state at different time-periods and under varying conditions. At the root of the method used in assessing structural strength lie research works of academician P. A. Rebinder and representatives of his school. The measurement of the structural strength of quick-setting plugging mixtures may be based on the method of the conical indenter plastometer, whereby a conical indenter is allowed to penetrate the thickening plugging mixture and the penetration depth of the indenter under a given load is measured.

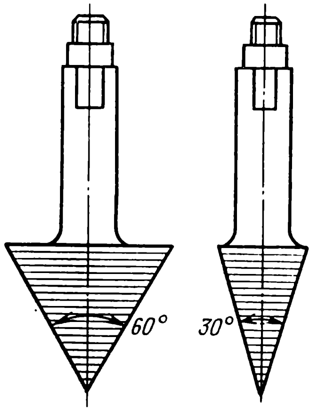
According to P. A. Rebinder, the plastic strength P_m (in Pa) of a system is given by the following relation:

$$P_m = k_\alpha (F/h^2) \qquad (50)$$

where k_α is a coefficient depending on the shape of the indenter (α is the cone angle), F is a constant (adopted) load on the indenter, N, and h is the penetration depth of the indenter, cm.

Some Soviet petroleum research bodies measure the plastic strength of the structure of various plugging mixtures by using the standard Vicat apparatus, but instead of the needle a set of conical indenters has been proposed. Figure 96 shows conical indenters with different angle α and of different mass. This set of conical indenters enables one to evaluate different structural states of plugging mixtures, their plastic limit from 0.005 to 0.05 MPa. Figure 97 presents two curves that characterize changes in the plastic strength of a quick-setting (curve *1*) and a slow-setting (curve *2*) plugging mixture.

The cited data are true for static conditions. A substantial influence on the structurization of plugging mixtures, especially quick-setting, exerts the process of agitation (movement) of the mixture. It has been established that the process of movement tends to retard the setting and thickening of slurries and mixtures. But one has to consider the duration of the movement (agitation) of the slurry and the ambient conditions (mainly temperature). Thus, if in a concrete situation agitation terminates at an early stage of coagulation structurization, the setting of a plugging mixture may be even accelerated. With a long-term and intensive agitation, a slurry or a quick-setting mixture may remain mobile, though around individual crystallization centres there will be in evidence a strongly marked thickening and setting of the mass.



(*a*) (*b*)

Fig. 96. Conical indenters
(*a*) with a cone angle of 60°; (*b*) with a cone angle of 30°

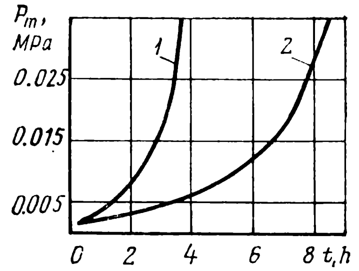


Fig. 97. Plastic strength of plugging mixtures as a function of time at 22 °C

Laboratory Work No. 10

I. *Composing a quick-setting mixture* (*QSM*).

Intermingle cement with gypsum in the ratio 5 : 1 and mix the whole with water. Pour the stirred mixture into a Vicat ring to determine its setting time.

II. *Measuring the plastic strength of quick-setting mixtures by using a plastometer.*

Replace the needle in a standard Vicat apparatus for determining the setting time of slurries with a conical indenter. Pour the test cement mixture in a ring set up on a plate and smeared with petroleum jelly; cut off the excess. Bring the point of the indenter to the surface of the cement mixture and let the indenter fall. Determine the penetration depth of the indenter into the mixture every 5 to 10 minutes in the case of normal or slow-setting plugging mixtures and every 1 to 2 minutes in that of quick-setting mixtures. By using formula (50), determine changes in the plastic strength of the cement mixture. Plot the changes in the plastic strength of the cement mixture against time.

Chapter 13

Special Plugging Cements and Mixtures

13.1. Corrosion-Proof Plugging Cements

By its very nature, cement stone proves thermodynamically unstable in aqueous solutions containing various ions, especially when it is acted upon by high temperatures and pressures. In the end, it must disintegrate and lose its plugging properties.

The stratal waters in most deposits of the Soviet Union contain mainly sodium and potassium chlorides. It is known, however, that the brine waters of many reservoirs in Uzbekistan, the Volga region, Bashkiria, the Ukraine and North Caucasus have high concentrations of sulphate ions, magnesium ions, hydrogen sulphide, and carbon dioxide. At high temperatures and pressures, these brine waters, particularly the ones carrying hydrogen sulphide, are capable of decomposing some kinds of plugging materials over a period of one year and even in less time. Thus, for instance, the stratal waters of the Urta-Bulak, Dengiz-Kul and other areas (Uzbekistan) contain considerable amounts of hydrogen sulphide. Besides hydrogen sulphide, the waters of the Dengiz-Kul deposit comprise also magnesium and sulphate ions in scores of thousands of milligrams per litre. The cement rings of the wells in these deposits are susceptible to quick disintegration if special measures are not taken to protect them against corrosion.

But the thermodynamic instability of materials in the given environment does not necessarily mean that they are invariably doomed to rapid destruction. The rate of corrosion to which materials are exposed under different conditions varies over an extremely wide range and the corrosion resistance of the cement ring can be adjusted through proper selection of plugging materials and their additives.

With the so-called active mineral additives containing silicon dioxide introduced into cements, the mean basicity of the hydrosilicates drops to 0.8-1.2. At an ambient temperature of 150 to 160 °C, low-basicity hydrosilicates of the so-called tobermorite group or of the CSH(B) composition (0.8 to 1.2) and $CaO \cdot SiO_2 \cdot nH_2O$ develop, whereas at higher temperatures xonotlite $Ca_6Si_6O_{17}$ is formed.

At temperatures of 50 to 70 °C, substances carrying noncrystalline, amorphous silica (tripoli, opoka, diatomite, Si-stoff pumice, tuffs) play the role of active siliceous additives. The first four additives display a very high activity in combining calcium hydroxide which is released during hydrolysis of cement, since they are porous materials with a huge surface of capillary pores.

At higher temperatures, common quartz sand also displays an appreciable activity in interacting with lime. The finer the grinding of the sand and the smaller the size of its grains, the quicker the sand binds calcium hydroxide that is released during hardening of cements, lowering thereby the basicity of hydrosilicates and calcium hydroaluminates. The larger the grain size of the quartz sand added to cement, the higher should be the temperature and the longer the time needed for the sand to be completely bound by calcium hydroxide of the cement.

The composition of the products formed during hardening of slags does not generally differ from the composition of cement hydrates. The basic distinction between slags and portland cement consists in a lower content in the former of calcium oxide and a higher level of silicon dioxide. Cement, for example, contains from 60 to 65 % of CaO and from 20 to 25 % of SiO_2 and slags, 20 to 50 and 30 to 50 %, respectively.

As a result, slag stone contains less calcium hydroxide than does portland cement stone.

One of the types of cement stone corrosion can be corrosion due to the lixiviation of calcium hydroxide.

All other things being equal, the greater the concentration of lime in the pores of cement stone, the higher the rate of lixiviation. The higher the concentration of Ca^{2+} and OH^- ions in the leaching medium, the slower the rate of lime lixiviation, and that is why "soft" waters with a small content of calcium salts are more aggressive than "hard" ones.

Sodium chloride tends to significantly raise the solubility of lime and therefore mineralized waters carrying this salt possess greater leaching capacity than fresh waters. Increasing temperature adds to the mobility of calcium ions, i. e., increases the diffusion coefficient, or diffusivity, and therefore as the temperature goes up, so does the rate of the lixiviation-induced corrosion. Diffusivity has appreciable influence on the lime leaching rate. The latter is determined by the permeability of cement stone and its structural characteristics. The denser the cement stone and the smaller the radius of its pores, the less marked is diffusivity. It may be brought down by reducing the water-cement ratio of the plugging mixture, adding to it high-molecular agents, such as hypan, CMC, etc.

In contrast, lightened cement slurries (with additions of clay) of a high water-cement ratio have pores of a larger size and consequently a higher diffusivity.

Slag binding agents are characterized by a low concentration of lime in their liquid phase and a moderate porosity, and for this reason, lime is poorly leached from them.

If a medium surrounding cement stone contains substances that form with calcium hydroxide little-soluble or little-dissociable chemical compounds, the concentration of lime therein will continue to be at a very low level for as long as these substances are still available. This phenomenon is seen to occur, for example, under the effect of magnesium sulphate solutions:

$$Ca(OH)_2 + MgSO_4 + 2H_2O = Mg(OH)_2 + CaSO_4 \cdot 2H_2O$$

As a result of this reaction, there emerges magnesium hydroxide, whose solubility at room temperature is but some hundredths parts of a gram per litre of water, and gypsum with solubility in water of about 1.9 g/l at room temperature. As a consequence, the action of magnesium sulphate solutions can cause complete destruction of cement stone. Magnesium chloride solutions produce a similar but a weaker effect.

An important feature of the process of magnesium-induced corrosion is the formation of little-soluble magnesium hydroxide which deposits within the corrosion zone in the pores of cement stone, clogs them up and thus slows down the penetration of new portions of the aggressive solution deep into the stone. The colmatage part played by the layer of the little-soluble corrosion products (magnesium hydroxide and gypsum in the case of magnesium sulphate) is determined by the layer density which in turn depends upon the lime content in the pores of cement stone. From this it follows that the higher the basicity of the binder, i. e., the lime content therein, the more compact appears to be the layer of the corrosion products, the stronger it inhibits the advance of the corrosion front deep into cement stone and the greater is the magnesia resistance of the latter.

Introduction into cement materials of active mineral additives (diatomite, opoka, pumice, tuff, clay, and ground quartz sand at elevated temperatures) reduces the content of lime and lowers the magnesia resistance of the binders in proportion to the quantity of the active additive. Pozzolan cements which contain substantial amounts of active mineral additives are liable to disintegrate in magnesia solutions much faster than pure portland cement.

Therefore, in media with a sizeable concentration of magnesium ions, application of lightened cement slurries with active mineral additives is inadmissible.

The calcium chloride content of slag cements is lower than that of portland cement by a factor of 1.5 on the average, but these two kinds of cement differ but slightly in magnesia resistance. The main reason for this is the fact that during decomposition of slag hydrosilicates there develops a considerable amount of silica which is distinguished by a greater density owing to its special structure and thus exerts a significant colmatage action. But in this case too, it is expedient that in conditions of magnesia aggression slags of elevated-basicity should be employed. However, any additions of clays and active mineral substances to slags are inadmissible. Magnesium chloride is much less aggressive than is magnesium sulphate.

In oil and gas fields, one of the most common types of corrosion attended by the formation of little-soluble sediments is that due to hydrogen sulphide. In cases of the hydrogen sulphide-induced corrosion, the development of little-soluble calcium, aluminium, and iron sulphides is observed:

$$Ca(OH)_2 + H_2S = CaS + 2H_2O$$
$$2Al(OH)_3 + 3H_2S = Al_2S_3 + 3H_2O$$
$$2Fe(OH)_3 + 3H_2S = Fe_2S_3 + 3H_2O$$

Iron sulphide forms most vigorously and therefore, to raise the resistance of binders to attack by hydrogen sulphide, one should limit the content therein of iron, manganese, and other heavy metal oxides that form stable sulphides.

The formation of little-soluble compounds, accompanied by an increase in their solid phase volume is the most complex type of chemical corrosion affecting cements. This is exemplified by the action of sulphate solutions, for instance, of calcium or sodium sulphate, on portland cement stone. Into the composition of the hydroaluminate phase of the portland cement enter calcium hydroaluminates which interact with calcium sulphate and form a compound (complex) salt — calcium hydrosulphoaluminate. Since during formation of this salt much water enters its solid phase, the volume of the crystals increases several times. The calcium sulphoaluminate crystals, while growing in size and volume, cause high internal stresses in cement stone, which eventually leads to fracturing and complete disintegration of the stone.

Several methods of combating the sulphoaluminate aggression are known. The simplest among them is introduction into portland cement of active mineral additives reducing the concentration of calcium hydroxide, such as tripoli, diatomites, opoka, baked kaolinite clay, tuff, pumice, etc.

Another way of keeping down the hydrosulphoaluminate aggression is limiting the content of tricalcium aluminate in the cement. Sulphate-resistant cements should contain no more than 5 % of this mineral.

The presence in the aggressive medium, besides sulphates, of a considerable amount of chlorides, and the more so, the prevalence of the latter (this being commonly observed in formation waters), diminishes the adverse effect of the sulphoaluminate aggression. Worthy of notice is also the thermal instability of calcium hydrosulphoaluminate.

In the Soviet practice of cementing wells, use have found corrosion-resistant plugging cements among which are pozzolan, sand, and slag-portland cements. In some cases, high alumina and carbonate cements are also employed.

Of major importance is the correct proportioning of corrosion-resistant plugging cements in concrete conditions of cementing jobs, with due regard for the chemical composition of stratal waters.

In cementing of wells with a temperature of 50 to 70 °C in the lower portion of their bore and with an SO_4^{2-} ion content in stratal waters of approximately 500 to 1000 mg/l and more, it is recommended that sulphate-resistant cements with a low (less than 5 %) content of tricalcium aluminate should be used.

If then the content of Mg^{2+} ions in formation waters does not exceed 1000 mg/l, an addition to cements of a limited amount (10 to 15 %) of clays, opoka, tripoli, and chalk and also up to 50 % of ground quartz sand is admissible.

When the temperature in the lower portion of the well bore is 90 to 100 °C and higher, cement-sand and slag-sand compounds are considered to be the best material.

With the level of Mg^{2+} ions in stratal waters exceeding 1000 mg/l and the temperature near the bottom of the well bore up to 100 °C, recommended for use are portland cement with a limited content of tricalcium aluminate without silica-containing additions and also slag and slag-cement mixes.

Addition of siliceous materials, especially finely ground ones, tends to impair the magnesia resistance of cements and slags. In the event of magnesia aggression, one should minimize the water-cement ratio of plugging mixtures.

In the case of hydrogen sulphide aggression, the use of slag-sand and cement-slag-sand mixtures with an addition of a finely ground sand is recommended.

Cements and slags should contain a minimum of iron and aluminium oxides. Of prime importance here is to secure a high density of cement stone and for this reason, it is essential to keep to a minimum the water-cement ratios of plugging mixtures. In this case, it is most opportune to employ diatomites, opoka, and other siliceous materials carrying a minimum of iron and aluminium oxides.

Should there arise a need for adding weight to plugging mixtures in the event of hydrogen sulphide aggression, it is then advisable not to apply iron compounds, such as magnetite, hematite, and others, but make use of barite.

In the event of carbon dioxide aggression, one should make use of portland cement, slag-cement and slag mixtures. The addition of siliceous materials is to be limited.

In the absence of the aforementioned aggressive agents in stratal waters at a temperature above 80 to 100 °C, it is mainly thermal corrosion of cement stone that is possible, for lixiviation corrosion in its pure form does not apparently present any great danger in this case. Moreover, measures that provide for high thermal resistance of cement stone also raise its resistance against corrosion due to leaching. With the ambient temperature exceeding 100 °C, the use of the portland cement-based mixtures without active siliceous additives is not recommendable. The best additive is 40 to 50 % of ground quartz sand to portland cement, or 30 to 50 per cent of it to blast-furnace slag. At 120 to 130 °C and higher, the utilization of unground quartz sand is also possible. In conditions of high temperatures, one should employ cements, slags, and additives with a minimum content of aluminium oxide.

13.2. Expanding Plugging Cements

Several types of expanding plugging cements are used in the construction and oil and gas industries. Their use in cementing of wells is restricted. The expansion of most expanding cements is caused by calcium hydrosulphoaluminate crystals that develop and grow in size during their hardening. The expansion of magnesium oxide on hydration is also used to the same end.

The formulation of expanding plugging cement must be selected in such a way as to allow the growth of crystals in the compounds to continue till a definite point at which the structure of the solidifying stone attains its maximum strength. Should an intensive expansion go on after the stone has acquired a high strength, the stone may become subject to disintegration. Even insignificant departures from the selected composition of the cement may result in destruction of the stone.

The majority of mixtures prepared of expanding cements have accelerated setting rates and quickly harden at elevated temperatures. Under the effect of high temperatures and pressures, the alumina cement and calcium hydrosulphoaluminate, the principal constituents of expanding plugging cements, are unstable.

The widest application in the USSR have found expanding water-tight and expanding gypsum-alumina cements. The former is quick-setting, it being prepared by intermingling or joint grinding of high alumina cement, hemihydrate gypsum, and a specially treated calcium hydroaluminate.

Mixtures made of expanding gypsum-alumina cement consisting of finely ground, high alumina blast-furnace slags and natural dihydrous gypsum in the ratio 70 : 30 set at a slower rate. Because of the sulphoaluminate decomposition, their use is limited to temperatures of 70 to 80 °C.

In this country, application find expanding cements of the following compositions:

(a) portland cement — 60 to 65 %, alumina cement — 3 to 6 %, dihydrous gypsum — 5 to 10 %, hydraulic additive — 20 to 25 %;

(b) clinker — 80 to 82 %, unslaked lime — 15 %, dihydrous gypsum — 3 to 5 %; stone expansion in this case is 3 to 4 %;

(c) portland cement — 70 %, diatomite — 30 %, gypsum-alumina cement — 15 %; stone expansion here is up to 1 % at a water-cement ratio of 0.8.

In the practice of cementing wells, some application have found expanding plugging cements composed of 80 to 90 % of portland cement and 10 to 20 % of gypsum-alumina cement. The stone expansion is up to 1 %.

To lengthen the setting time of slurries prepared from expanding plugging cements, the latter may be treated with some retarding agent.

13.3. Gypsum as a Plugging Material

The application of gypsum as a plugging material has become known comparatively recently. It has sometimes been used as a lost circulation material in drilling of wells. High-strength gypsum is now employed also in cementing surface casing and, sometimes, intermediate casing strings.

Several modifications of gypsum are known, namely dihydrous gypsum, $CaSO_4 \cdot 2H_2O$, hemihydrate gypsum, $CaSO_4 \cdot {}^1/_2H_2O$, and anhydrous gypsum, $CaSO_4$ — a soluble (artificial) anhydride or insoluble natural or artificial anhydride.

Hemihydrate gypsum is the basic component of construction gypsum. It is also contained in high-strength gypsum. The density of hemihydrate gypsum is 2.60 to 2.75 g/cm^3. The method of obtaining this kind of gypsum determines its composition. With one of them, the resulting product is construction (β-hemihydrate) gypsum and with the other, high-strength (α-hemihydrate) gypsum. The salient feature of the latter is the high strength of the gypsum stone during the first hours of its maturing.

When mixed with water, hemihydrate gypsum changes into dihydrous (dihydrate) gypsum — a high-strength stone distinguished by its microcrystalline structure.

Because of their rapid setting, high-strength gypsum slurries are not used without setting retarders in cementing of wells.

An exception to this are only individual cases of cementing conductors or test-pits. The initial set of high-strength gypsum slurries does not take more than 10 to 20 minutes. In ordinary conditions, neutralized black contact (NBC), sulphonol, and spent sulphite-alcohol liquor produce a good retarding action on the gypsum slurries. Their retarding action is based on the formation of a surfactant film around the gypsum grains, which prevents the access of water into the bulk of gypsum. Retarders of another group, which includes inorganic substances, reduce the solubility of gypsum and thus slow down the setting of gypsum. Among these retarders are borax, sodium tripolyphosphate, sodium hexametaphosphate, superphosphate, etc.

The retarders are preliminarily dissolved in the water with which gypsum is mixed. The least desirable as a retarder is superphosphate whose proportion must be pretty high; it causes foaming of the slurry.

Temperature has a substantial influence on cutting down the setting time of gypsum slurries. The accelerating effect of temperature is manifest at up to 50 °C, following which its action is reversed and the setting is retarded. The retardation is roughly proportional to the rise in temperature. At a temperature of about 80 °C, a hemihydrate gypsum slurry does not set at all. Hence, the field of high-strength gypsum application as a plugging cement is confined to situations where the ambient temperature does not go beyond 50 °C.

Initially, the water loss of a pure gypsum slurry is rather great, but the rapid progress of its setting makes it impossible to study this process in greater detail. In the presence of retarders, the water loss of a gypsum slurry increases parallel with the rise of pressure differential.

The bending strength of gypsum stone reaches 2.5 to 3 MPa after a few hours (3 to 5). A significant effect on the strength of the stone have the type and proportion of the setting retarder introduced into the slurry. Thus, an addition of 9 % of a retarder brings, after 3 hours of hardening, the bending strength of the stone down to zero.

Hardening of a gypsum slurry is attended by liberation of heat. According to P. P. Budnikov, the hydration heat, i. e., the quantity of heat liberated by 1 g of gypsum, amounts to 103.2 J/g. Typical of gypsum slurries is the liberation of virtually the entire heat during the first hours of setting and hardening.

In hardening, gypsum slurry and stone expand. According to Yu. M. Butt, the expansion of not yet hardened gypsum slurry equals 1 %.

Gypsum is an air-slaked binding agent. The insufficient water-resistance of gypsum stone is due to the solubility of calcium sulphate in water, which is more than 2 g/l at 20 °C. In most mineralized waters, gypsum stone becomes less water-resistant and this limits its use in the oil-field practice.

The water-resistance of gypsum can be raised by adding to it up to 50 % of slag.

13.4. Hydrophobic Cements

After a long-term storage, especially during autumn and winter months, the properties of plugging portland cement deteriorate, *viz.*, cement gets lumpy, the setting time of cement slurry lengthens, its viscosity increases, and the mechanical strength of cement stone declines. Long storage in damp surroundings sometimes results in that the whole of the cement, kept in ordinary paper sacks, becomes transformed into a stone-like body.

Reduction of the plugging cement hygroscopicity and, consequently, its better preservation can be achieved by treating it with additives that render it hydrophobic (water-repellent). In the case of construction cements in use among such water-repellent additives are acidol (a mixture of water-insoluble naphthenic acids), naphthenate soap, oleic acid, fatty acid residues, oxidized petrolatum, and others. The additives are usually introduced into the mills during grinding of cement clinker.

The properties of hydrophobic cement that make it advantageous both technically and economically include the following:

(a) water-repellent characteristics facilitate fragmentation of cement clinker, i. e., they allow one to intensify the grinding of cement and thus increase the efficiency of grinding departments at the mills;

(b) cement flour that is rendered hydrophobic is practically non-hygroscopic and, therefore, it is capable of preserving its activity during a long period of storage even in damp surroundings.

Plugging cements should be readily wettable and, at the same time, have low hygroscopicity.

Water-repelling additives must not reduce the strength of cement stone, nor increase its permeability or raise the viscosity of the slurry; they lengthen the storage life of cement by reducing its hygroscopicity.

The hygroscopicity of plugging cements can be reduced by introducing into them (at the time of grinding) aminoalcohols, such as monoethylamine, $NH_2C_2H_4OH$ (up to 0.5 %), diethanolamine (0.25 to 0.5 %), and triethanolamine (0.25 to 0.5 %).

13.5. Oil-Cement Slurries

Petroleum-cement slurries that consist of cement and petroleum or diesel fuel occupy a particular place among plugging mixtures. The main advantages of such mixtures are their inability to set in the absence of water and high strength of the stone they yield, which is because of their interaction with an insignificant quantity of water (20 to 25 %). When penetrating into water-conducting channels the mixture quickly gets thick and, while giving off petroleum (or any other base product, such as diesel fuel, kerosene, etc.), it interacts with water and thus yields a firm stone.

When a petroleum-cement slurry is mixed with water, its mass very quickly loses its mobility, turns into lumps and a stone, releasing nearly all of the petroleum product contained therein. To increase the mobility of the mixture and achieve better washing away of the petroleum product, use is made of highly effective surfactants, *viz.*, stillage residue of ortho-ethylsilicate, cresol, dimer, acidol, and calcium naphthanate. Cresol ($CH_3C_6H_4OH$) employed in the oil industry as a selective solvent is more readily available.

An elevated content of a petroleum product and surfactants make the mixture more mobile, while a long-term storage of a petroleum-cement slurry together with diesel fuel and in the presence of the stillage residue (SR) reduces its fluidity. The quantity of the petroleum product is habitually taken at 40 to 50 % by mass of cement.

As concerns its action on petroleum-cement slurries, cresol differs somewhat from the stillage residue, though in many respects their properties are the same. With addition of one percent of cresol, the mobility of a petroleum-cement slurry increases, while with larger amounts of it the mobility declines. During the first 1 to 8 hours of keeping the slurry, cresol tends to increase somewhat its mobility. During a long storage (up to 5 months) of the slurry, into evidence comes its slight dilamination and liberation of fuel from it. With cresol, petroleum-cement slurries display a greater mobility at rising temperature. When intermingled with hot water, petroleum-cements rapidly get thick to form a non-flowable mass and, as they combine with water, they intensively liberate the petroleum product. When combined with 30 to 35 percent of water, the slurry gets rid of 90 to 95 per cent of the petroleum product, turns into a thick mass and then into a stone.

Petroleum-cement slurries (without water) fail to set at temperatures above 200 °C and pressure of 70 MPa. The replacement of 20 % of the petroleum product with water results in that the slurries rapidly get thick already at 120 °C and a pressure of 30 MPa and then set in 20 to 30 minutes.

Petroleum-cement slurries with an accelerator added may serve as a base for preparation of quick-setting mixtures for controlling lost circulation in wells. In the absence of water, such slurries fail to set. Water contributes to their rapid thickening and the accelerator — to setting. Soda ash, gypsum and other materials may serve as setting accelerators. In individual cases, aggregates (fillers), such as sand and clay, may be introduced into petroleum-cement slurries to improve their properties.

Both the ordinary (aqueous) and petroleum-cement slurries gain access into formations only along crevices and channels and virtually fail to penetrate the bed pores.

A partially set petroleum-cement mass becomes subject to an intensive disintegration under the effect of sulphuric acid. The latter, while reacting with the petroleum product, displaces it and, having gained access to the cement particles, interacts with them. This results in a vigorous self-heating of the mass, accompanied by evolution of gases and by the formation of dihydrous (dihydrate) gypsum, calcium sulphoaluminate, and of other sulphates. The emergence of these compounds is attended by a significant increase in the volume of the cement bulk and is instrumental in bringing about its rapid destruction.

Petroleum-cement specimens quickly disintegrate under the effect of a 10- to 25- % sulphuric acid solution. The intensity with which a petroleum-cement mass disintegrates depends upon the quantity of water that has reacted with the cement, the concentration of the acid, and conditions attending the reaction.

Hydrochloric acidizing may be practised in wells where aquifer partings lie close to the oil measures. Within the latter, the disintegration of a partially set petroleum-cement stone will take an intensive course, whereas within aquifer partings containing an elevated quantity of water, a plug of good quality must form.

13.6. Organic and Organic-Mineral Materials for Cementing Wells

The mineral-binder-base plugging mixtures that are now being widely used often fail to provide for reliable isolation of formations.

The problem of high-quality well cementing remains very pressing. Solving this problem successfully requires such plugging materials as would be free from the shortcomings typical of mineral binders. Such materials may be plugging mixtures based on macromolecular compounds.

The most valuable properties of man-made (synthetic) materials have been revealed comparatively recently. The application field of these materials grows wider year after year. Plastic materials have a low density, they are resistant to attack by various corrosive media, and many of them feature a high adhesion to metals and rocks. That is why they find an ever increasing application in many fields of engineering. For example, construction engineering makes wide use of polymers as additives to cements to improve their quality.

In most general terms, the problem on the development of plugging polymer materials may be formulated as follows: a system suitable for plugging jobs must have a low initial viscosity (no more than 0.5 Pa s) and adjustable hardening time and must harden into a shrinkage-free stone of definite physical and mechanical properties.

The possibilities of macromolecular chemistry, which can solve problems on transforming liquid systems into solids that are expected to be used as a plugging stone, are great. Hard, monolithic products can be formed from liquid ones by way of one of the following three main reactions: polymerization and copolymerization, polycondensation, and macroanalogous conversions.

Research work carried out at one of the Soviet scientific research institutes has made it possible to obtain new plugging materials by using the method of macroanalogous conversions. Curable polyvinyl chloride (PVC) is the most interesting material of this class and has received most study.

The PVC-base plugging material, while possessing all the advantages of polymer materials, is free from the shortcomings characteristic of portland cement and slag stone. Its formulation includes powdered PVC Grade E-62, dibutylphthalate (DBP), *o*-xylene, kaolin, and anhydrous zinc chloride. Analysis has shown the following formulation to be most rational:

(a) the proportion (by volume) of PVC — 5 to 8 % (this percentage corresponds to the maximum strength of stone and adequate thickening time which shortens with increasing concentration of PVC);

(b) the proportion (by volume) of zinc chloride — 0.7 to 1.1 % (this percentage corresponds to medium thickening time; the strength of stone depends but weakly on the concentration of zinc chloride);

(c) the proportion (by volume) of kaolin — 18 to 20 % (this percentage corresponds to medium thickening time and strength of stone and gives adequate standard fluidity cone test of the composition);

(d) the proportion (by volume) of DBP — 18 % (this percentage corresponds to the maximum strength of stone and maximum thickening time);

(e) the maximum strength of stone at a sufficiently long thickening time is observed at temperatures of 80 to 135 °C.

PVC-base plugging mixtures can be successively used in cementing wells with bottom-hole temperatures up to 135 °C.

An essential feature of the PVC-base plugging stone is its ability to swell by 15 to 20 % in an aqueous medium and to shrink by 20 to 28 % in a hydrocarbon medium.

Considering the above advantages, PVC-base plugging mixtures can be employed for cementing wells and controlling loss of circulation in them and also for selective isolation of formations in recementing producing wells.

Plugging materials based on hypan (hydrolyzed polyacrylonitrile), polyvinyl alcohol, carboxymethyl cellulose, and polyacrylamide have also been obtained in a similar way.

Polymer-base plugging materials possess a number of advantages over mineral-binder-base materials, namely:

(1) low density that can be adjusted over a wide range;

(2) adjustable thickening time;

(3) hardenability of liquid-phase filtrates; where such filtrates penetrate the wall mud or asphaltic cake and permeable rock, they harden there and thus provide for the continuity of the plugging stone, the cake, and the rock;

(4) possibility of ensuring adhesion of the plugging stone to the casing string metal and to rocks;

(5) high sedimentation stability;

(6) absence of contraction processes in the course of hardening;

(7) practically absolute impenetrability of the plugging stone in all cases;

(8) inertness of polymer-base stone sludge to clay drilling muds;

(9) high inertness of polymer-base plugging materials to well fluids.

Polyolefine-cement compositions. Here we consider mainly polyethylene. This polymer is a thermoplastic material. Its second order transition point is 115 °C, melting point 137 °C, tensile strength 24.5 MPa, elastic modulus 210 MPa, and elongation at rupture 500 %. At temperatures above 130 °C, the C-H bond of polyethylene can dissociate and the polymer in these conditions can cross-link to form a three-dimensional structure.

The presence of colloidal polyethylene in cement stone improves, sometimes in a fairly good measure, some of its properties, such as plasticity and deformability, bending and breaking strength, corrosion resistance, and permeability to water.

In polyolefine-cement compositions, polyethylene is introduced in the form of solid particles or in the form of suspension of polymer powder in water or in some other liquid. Cement stone reinforced with polyethylene filaments has an improved impact strength.

Water-base polyethylene suspensions can be successfully used to modify the properties of slag stone. Polyethylene-slag compositions can be recommended for testing in "hot" oil and gas wells.

Polyvinyl chloride-cement compositions. Polyvinyl chloride is a white, amorphous polymer having a high surface hardness (15 to 16 HB). The macromolecules of polyvinyl chloride are long polycarbon chains in which 75 % of the bonds not spent to form the backbone are occupied by hydrogen atoms and 25 % by chlorine atoms.

The second order transition point of PVC is 81 °C and its melting point is 212 °C, but already at 120 °C, PVC starts decomposing, giving off hydrogen chloride.

By using vinyl chloride-vinyl acetate copolymers, a shrinkage-free plugging stone has been obtained having a high moisture resistance.

Of substantial interest is the use of polyvinyl chloride-polyvinyl acetate latexes. These latexes do not coagulate under the effect of polyvalent cations and can be successfully used for modification of cement slurries. The introduction of the latexes into the slurries improves their mobility and pumpability and reduces the permeability of cement stone by a factor of several score. The use of a copolymer of vinyl chloride and an unsaturated acid, its anhydride, or amide improves the elasticity and homogeneity of cement stone.

The above data indicate that cement compositions containing PVC copolymers can be recommended for cementing wells with not very high bottom-hole temperatures (50 to 60 °C) in conditions of acid water corrosion.

Polyvinyl acetate-cement compositions. At temperatures below 80 °C, polyvinyl acetate (PVA) is a glass-like material. When heated above this temperature, it softens and acquires plastic properties. The material retains these properties until heated to a temperature of 120 to 130 °C. At temperatures above 130 °C, PVA decomposes to form acetic acid.

Polyvinyl acetate slightly swells in water; it is insoluble in gasoline and kerosene, but dissolves readily in polar organic liquids and aromatic hydrocarbons; its adhesion to silicate materials is high.

The low resistance of PVA to hydrolysis in the alkaline medium of cement slurry prevents the plugging stone to retain its newly acquired properties for any lengthy period of time. The process of hydrolysis is sharply accelerated in a moist, hot medium.

In the course of hydrolysis, PVA gradually converts to polyvinyl alcohol. When the degree of conversion reaches 60 %, polyvinyl alcohol, being soluble in water, starts leaving the cement stone under the effect of hydrostatic pressure differential (if any) or as a result of osmosis.

The hardening of PVA-cement compositions is a combined process of hydration hardening of cement clinker and drying of polymer dispersion. Addition of calcium chloride favours the formation of PVA-doped cement stone. The amount of PVA added to cement may vary from fractions of a percent to 20-30 %, up to 50 % PVA being used to prepare cement glue.

The introduction of PVA into cement slurry lengthens the initial set period of the latter, and with the amount of the polymer added not exceeding 30 %, this lengthening is proportional to the polymer content.

Polyvinyl acetate-cement compositions possess good plugging properties, but the low resistance of PVA to hydrolysis and reduced strength of the compositions in moist media limit the possibilities of their use. The compositions can be used for a temporary isolation of formations in fighting loss of circulation and in workover jobs.

Polyvinyl alcohol-cement compositions. Polyvinyl alcohol (PVAlc) is the only synthetic aliphatic-series hydroxl-containing polymer that is now being produced on a sufficiently large scale.

PVAlc is one of the few water-soluble polymers, which facilitates its introduction into cement slurry.

The high degree of polymerization of PVAlc (its molecular mass is 10^6 and more) and the expanded shape of its macromolecules in an aqueous medium enable it to effectively reduce the water loss of cement slurry. Addition of 0.4 % PVAlc reduces the water loss from 40 to 20 ml in 30 min, while adding 0.8 % PVAlc reduces it to 5 ml in 30 min.

Adding 1 to 2 % of PVAlc to a cement-sand (1 : 2) mixture improves the compressive strength of cement-sand stone, and increasing the content of PVAlc to 10 % makes the slurry highly adhesive, so that it forms a strong bond with old cement stone.

Under certain conditions of service, the properties of cement stone can be improved by adding PVAlc, since the latter reacts with many substances, boron compounds in particular, to form water-insoluble complexes. Introducing complexing agents into cement slurry will help retain PVAlc in the stone. Another way is to add PVAlc to cements that are intended for use at temperatures above 100 °C. Under the effect of such temperatures, especially in an alkaline medium, PVAlc cross-links into a three-dimensional structure forming a very strong framework. Investigations have shown that PVAlc in dilute solutions can be made to cross-link into a three-dimensional structure at temperatures of 30 to 100 °C by treating the polymer with a reduction-oxidation system containing ammonium bichromate and sodium thiosulfate.

Polyacryl-cement compositions. This group includes compositions based on cement with additions of polyacrylic acid, its salts, or polyacrylamide. These polymers are readily soluble in water. At the same time, the parent monomers easily enter into copolymerization reactions with many polyfunctional monomers to form three-dimensional, water-insoluble products. The polymerization and copolymerization reactions, and also reactions between functional groups and cement clinker components, proceed at not very high temperatures and at a sufficiently high rate.

The organic component may be introduced into cement either in the form of a monomer which undergoes polymerization during the hydration of cement or in the form of a ready-made polymer.

The first method is the more popular and is used fairly widely in the mining and oil industries for consolidation of rocks and cementing of wells.

For copolymerization reactions, use can be made, in addition to acrylamide, also of acrylic and methacrylic acids and their water-soluble salts (alkali and alkali-earth). These additives make it possible to obtain cement stone of high early strength.

The second method of utilizing acrylic acid derivatives in organo-cement compositions, i. e., introducing a ready-made polymer into cement slurry, has also been put to use in the field. Introduction of small amounts of polyacrylamide into portland cement helps improve the initial mechanical strength of cement stone.

Aqueous solutions of polyacrylic acid (Grade K-4) can be used, in conjunction with a calcium chloride solution, to obtain quick-setting plugging materials. Calcium chloride is necessary to form an excess of calcium ions, otherwise the polymer, taking out of crystallization reaction the calcium ions contained in cement clinker, would deteriorate the properties of cement slurry and stone. These plugging mixtures have the highest density of all, and they set and harden, no matter what the corrosive effect of stratal waters.

However, small additions of polyacrylic acid and polyacrylamide solutions are more often used to retard the setting of cement slurries when cementing oil and gas wells in conditions of high temperatures, to reduce the water loss of cement slurries, and to make highly structurized gel-cements coagulate.

Addition of 0.015 to 0.025 % polyacrylonitrile (hypan) also retards the hydration of cement and reduces its water-requirement; if added in greater amounts, polyacrylonitrile shortens the setting time of cement slurries.

Polystyrene-cement compositions. Polystyrene is a transparent, hard polymer, very strong, and highly resistant chemically to attack by mineral reagents. It is insoluble in water, swells in saturated hydrocarbons, and dissolves slowly in aromatic and highly polar organic liquids. Among the advantages of polystyrene, worthy of notice is its high wear resistance which approximates that of a metal screen in sliding-contact friction and that of babbitt in rolling-contact friction.

Polystyrene shows a very weak tendency towards crystallization, this being due to the structural irregularity of its macromolecular chains. At temperatures below 100 °C, polystyrene is a glass-like substance, in the range 100 to 150 °C — a rubber-like polymer, and above 150 °C, it starts melting.

Polystyrene has been used to modify the properties of cement stone in one of the following four ways: by mixing cement with a water-base polystyrene suspension, by mixing cement with a water-base styrene emulsion, by introducing polystyrene fragments into cement slurry, and by impregnating ready-made cement articles with styrene, followed by its block polymerization.

Introducing 5 to 25 % styrene copolymers into cement slurry reduces its water-requirement and improves the mechanical strength of cement stone. Compositions based on cement with addition of divinylstyrene latex (Grade СКЗ-65ГП) possess a high resistance to atmospheric precipitation. The latex can be successfully used to protect cellular concrete against rainfall.

Introducing styrene into the mixing water is essentially a modification of the first method, and has a number of advantages over the latter (more uniform distribution of the polymer throughout the bulk of cement stone, higher quality of the resulting composition, etc.). It is, however, not without substantial shortcomings, the principal one being the need in subsequent polymerization of styrene.

Application also find some styrene derivatives (styrene acrylate in particular) in amounts of 1 to 5 %, which help to materially lengthen the setting time of cement slurries.

When sealing off lost circulation zones, the cement shield, should it prove to be permeable, can be impregnated with styrene by spotting a styrene bath in the region of the drilled-out cement bridging plug. A polymerization initiator must be added to styrene prior to its pumping into the well. The choice of the initiator depends on the temperature prevailing in the cement shield zone. It is advisable to use benzoyl peroxide for temperatures of 70 to 90 °C, *tert*-butylbenzoyl peroxide for 90 to 120 °C, and di-*tert*-butyl peroxide for 120 to 140 °C.

Phenol-cement compositions. Phenol aldehyde polycondensates (PAC) are among the most widespread and readily available synthetic materials. Therefore, investigations into their compatibility with cement have been carried out on a large scale. There are both acid- and alkali-curing PAC. Naturally, the former cannot form any three-dimensional structure in the cement-stone medium.

Analysis of the interaction mechanism between phenols and cement clinker components has shown that phenols react with calcium ions. The more polyatomic is the phenol, the longer and the more branched are the molecular chains it forms, and the higher the initial consistency of the cement slurry; the higher the phenol concentration, the greater is the number of calcium ions taken out of the hydration reaction of cement, and so the more the setting of the slurry is retarded. However, with a certain concentration of phenol and with formaldehyde present, the reaction of polycondensation starts to lead that of hydration, and the setting rate of the cement paste is thus increased. Starting with a 10- to 12- % concentration of phenol in the mixture, the phenol aldehyde polycondensate becomes capable of forming a three-dimensional structure of its own, which has a high mechanical strength. At the same time, the strength of the cement crystal complexes drops as a result of inactivation of calcium ions by the hydroxyl groups of phenols.

Experiments have shown that adding 25 % of resorcinolformaldehyde polymer to cement improves two to three times the strength of the cement stone and brings its permeability down to zero. Replacing monoaldehydes (formaldehyde, acetaldehyde) by polyaldehydes (glyoxal, polyoxymethylene) also improves the strength of the stone. The phenol-cement composition filtrates, both pure and filled with various inert fillers, are capable of hardening. When separating in the well, the filtrates penetrate the wall mud cake and, hardening there, strengthen it and improve its adhesion to cement stone. As testified by laboratory studies, the introduction of PAC into cement slurries improves the chemical stability of the resulting stone, reduces its permeability, improves its frost resistance and plasticity, etc. Also, this enables one to increase the proportion of cheap inert fillers in the slurries.

Polyamine-cement compositions. The group of polyamines includes macromolecular compounds containing amine nitrogen either in the side or in the main chain. As a rule, these compounds are readily soluble in water, have a high degree of polymerization, and are capable of interacting chemically with cement clinker components, calcium ions in particular. The strength of the C-N bond formed is not very high; it might be expected that this bond disrupts easily when the temperature of cement slurry increases in the course of cement hydration. The emergence of free calcium ions in the hydrated clinker grains must improve the strength of the crystal structure of cement.

Both polyamines and the products of their hydrolysis or thermal decomposition are of alkaline nature, which is essential in combining them with the highly alkaline cement slurries.

Polyalkyleneamines and polyalkylenepolyamines, when added in amounts of 0.05 to 8 parts by mass to 100 parts by mass of dry cement, substantially reduce the water loss of cement slurries, which allows one to recommend them for cementing wells. Although polyamines in general affect but slightly the setting time of cement slurry, it has been found experimentally that polyethylenepolyamine (PEPA) accelerates the setting of ordinary portland cements. Subject to further comprehensive investigations, it will be possible to recommend cements with additions of PEPA for cementing surface casing in permafrost regions.

Polyepoxy-cement compositions. The products of reaction between epichlorohydrin (chloropropylene oxide) and polyatomic alcohols have become very popular during the past 15 to 20 years.

Polyepoxy compounds are now being widely used in many branches of engineering. This is because of their many valuable properties. The high mechanical strength, isotropism, and corrosion resistance of polyepoxy compounds have made them attractive in connection with the necessity to impart similar properties to cements. Polyepoxy compounds are readily compatible with cements, irrespective of whether they are water-soluble or not. Addition of these compounds to cement slurry, as a rule, improves the properties of cement stone.

Investigations have shown that it is possible to obtain a lightweight stone (1.5 g/cm^3 in density) by modifying lightened cement slurries with triethyleneglycol and polyethylenepolyamine.

The principal factor limiting the appication of polyepoxy-cement compositions in cementing wells is their high cost.

Polyester-cement compositions. This group includes compositions containing saturated or unsaturated polyesters as their macromolecular component. In cases where cement is modified with an unsaturated polyester, the latter forms a spatial network of its own, which materially improves the properties of cement stone.

Commercially available polyesters are comparatively cheap and are readily compatible with cement slurry and stone. The compositions containing polyesters based on natural glycerines feature a very low gas permeability.

Saturated polyesters formed as a result of interaction between trimethylolpropane and adipinic acid improve the tensile strength of portland cement stone, but in conditions of elevated humidity, this polyester, if placed in an alkaline medium, is partially hydrolyzed, and so no increase in the strength of the stone is observed in this case. Almost all polyesters are resistant to attack by alkaline media. They pass their water-resisting property to polyester-cement compositions. In acid media (e. g., in oils with a high sulfur content), the compositions suffer destruction.

Introduction of large amounts of polyesters into cement slurries (e. g., up to 20 % of the total mass of cement, filler, water, and polymer in the slurry) allows high setting rates to be achieved at temperatures of 10 to 20 °C.

Polyester-cement compositions are disadvantageous in that they feature a considerable shrinkage, which is due to the high shrinkage (volume shrinkage — 5 to 8 %, linear shrinkage — 1.5 to 2.5 %) of polyesters in the course of polymerization and polycondensation.

Developed in this country are compositions containing 20 to 80 % cement and an unsaturated polyester, together with its copolymer (with styrene, for example). These compositions have an increased fluidity. When mixed with water, they quickly set, forming a strong, impervious stone. Such compositions can be used to seal off water-bearing horizons and to consolidate swelling clays prone to caving.

Furyl-cement compositions. Concurrent introduction into cement slurry of furyl pre-polycondensate and a curing agent (hydrochloric aniline), together with a hardener ($CaCl_2$), makes it possible to obtain cement concretes having elevated tensile and bending strength, frost resistance, and also resistance to attack by salts, oils, and gasoline. At the same time, the shrinkage deformations of the concretes are reduced, their crack resistance is improved, and permeability reduced approximately by 2 orders of magnitude.

Silicon-cement compositions. Organosilicon compounds are used to modify cements.

The large number of substances that are classed with organosilicon compounds differ widely in composition and properties, but have one feature in common, namely, they all contain silicon atoms in their main macromolecular chains, which makes them highly heat-resistant, chemically inert, hydrophobic, readily compatible with mineral fillers, highly adhesive, etc. These properties sharply improve the quality of cement stone.

At present, a number of organocement formulations have been proposed with organosilicon compounds as their ogranic component.

Organosilicon liquids (e. g., Grade ГКЖ-94) are introduced in an amount of up to 0.15 % into cement at the time of its grinding, this increasing the specific surface of the cement by 400 to 900 cm^2/g, i. e., by 20 to 25 %. The clinker grinding is in this case accelerated, and the strength of cement stone is improved by 10 to 20 %.

Solid organosilicon compounds, such as alkylpolyxiloxanes, may be added to the clinker in a finely dispersed form (1μ in grain size) in amounts of 0.1 to 4 %.

Water-insoluble alkyl (aryl) hydridethylsequioxanes, when added to cements, make the latter water-repellent.

Water-soluble ogranosilicon compounds may also be introduced directly into cement slurries. Their amount in this case may range from 0.025 to 10 %, sometimes reaching 100 % (by mass of dry cement). Addition of small amounts of organosilicon compounds to cement slurries improves their plasticity, while reducing their water loss and water-requirement. Some additives of this class, e. g., ethyl ether of ortho — silicic acid, improve the strength of cement stone by 15 to 20 % on the average.

Polysaccharide-cement compositions. Among polysaccharides, the widest application has found cellulose which is a reactive substance capable of interacting readily with some chemicals to yield derivatives with various substituents. A sodium salt of carboxymethyl cellulose, oxyethyl cellulose, sulfoethyl cellulose, etc. have recently been synthesized on the basis of cellulose. All these compounds contain acid functional groups, so that their action on cement by its nature is very much like that of organic acids.

Resin-cement compositions. Included in this group are such compositions wherein the role of polymer constituent is played by some natural resin. Classed with natural resins are tars of various origin, pitches, asphalts, coumarone-indene resins, paraffins, lignin, bitumens, etc. The properties of these substances are extremely diversified, and so they are used for a great variety of purposes.

Petroleum resins, obtained by catalytic polymerization of oil processing residues, are introduced into cement slurries in the form of emulsions which, like polyethylene, improve their pumpability, reduce the permeability of cement stone, and somewhat improve its elasticity.

Despite their relatively low cost, natural resins have as yet found but a limited application in preparing plugging mixtures.

Hardenable drilling muds for plugging jobs in wells. Modification of cements by additions of macromolecular compounds improves the quality of cement slurries and stone, but their principal disadvantages remain intact.

The question of the possibility of employing specially treated clay drilling muds as plugging materials has long since been discussed both in the literature and among pertoleum specialists. But to put this idea to practical use involves substantial difficulties.

To harden clay drilling muds, the latter must be treated with such substances as would be capable of forming, as a result of physical or chemical transformations, a three-dimensional, large cell structure with mud particles enclosed in its cells.

Analysis of the possible ways to harden clay drilling muds makes one believe that the most feasible method of obtaining hardenable clay drilling muds (HCM) is one providing for the formation of a three-dimensional polymer network in the clay mud medium. A three-dimensional polymer can be obtained through polymerization, polycóndensation, cross-linking, or vulcanization reactions.

In this country, investigations are now being conducted into the hardening of clay drilling muds by means of macromolecular compounds. Research results provide grounds to believe that the problem of hardening clay drilling muds has been solved in principle, so that now it is possible to turn clay drilling mud into stone in conditions of the well annulus.

Polycondensation of phenols and aldehydes in the presence of catalysts ($pH < 7$ or $pH > 7$) yields macromolecular products of stretched-out or branched structure. Of interest to the hardening of clay drilling muds are cases where three-dimensional products, known as resits, are formed. The character of resit formation depends on the specific structure of phenols and aldehydes, the mechanism of catalyst action, and the physical processes attending the chemical reaction.

Plugging compositions Grade ТСД-9, ТС-10, and ФРЭС were intended primarily for stabilization of producing formations. Recent investigations have shown that these compositions can be used as hardening (curing) agents for clay drilling muds in all types of isolation work in wells. These compositions contain shale-derived alkylresorcinols alkalized with an aqueous solution of caustic soda and stabilized with alcohols and glycols. To form a solid body when mixed with water, a mixture should contain 20 to 30 % TC-10 or 30 to 40 % ТСД-9, 25 to 70 % formaldehyde solution (to phenols), and water (balance).

Thickening time measurements, taken with the КЦ-5 and КЦ-4 Model consistometers, show that the consistency of the slurries remains unchanged for a certain period of time, following which the liquid systems rapidly turn into a solid body.

The thickening time of the slurries depends on their composition and temperature. The strength of the resulting stone is also dependent upon these factors, ranging from 0.5 to 1.2 MPa in compression and from 1 to 2 MPa in bending.

Introduction of ТСД-9 into a clay drilling mud treated with carbon — alkali reagent (CAR) yields a mass of low mobility. An easily pumpable mixture can only be obtained by mixing approximately equal volumes of ТСД-9 and a clay drilling mud with a density of 1.18 g/cm^3.

The TC-10 Grade composition does not cause thickening of clay drilling muds, and so it proves possible to formulate slurries containing up to 70 % mud. Tentative tests have demonstrated that the slurries have good physical, mechanical, and chemical properties.

In many regions of this country, the water used to prepare clay drilling muds contains considerable amounts of polyvalent salts. In some instances, the carbonate hardness of the water reaches $(80 \text{ to } 100) \times 10^{-3}$ mol/l.

Research results witness to the fact that slurries containing TC-10, a formaldehyde solution, and clay mud cannot be used at temperatures in excess of 40 °C.

In this connection, it proves interesting to investigate slurries containing not free, but combined formaldehyde in the form of formaldehyde-hexamethylenetetramine (a product of reaction between formaldehyde and ammonia) or in the form of a less reactive formaldehyde-furfural, which yield heat-resistant polycondensates. At certain proportions of TC-10, clay mud, formaldehyde solution, and hexamethylenetetramine or furfural, it proved possible to formulate technologically acceptable slurry compositions for use in the temperature range 40 to 80 °C. The clay mud used in the experiments was 1.18 to 1.20 g/cm^3 in density, 35 s in viscosity (as measured with the СПВ-5 Model apparatus), 5.3 to 6.0 cm^3/30 min in water loss (as measured with the BM-6 Model apparatus), and 5.8 to 10.0 Pa in gel strength. The bending strength of 2-day old stone specimens was in the range 1.0 to 2.0 MPa.

Investigations showed that as the density of the clay mud was increased, the strength of the specimens grew higher because of the reduction of the liquid phase content in the slurry. The filter loss of the slurry was by 30 to 60 % higher than that of the starting clay drilling mud, but the filtrate underwent polycondensation to form a hard plastic.

The permeability of specimens prepared from any compositions and under any conditions was found to be low, around (0.1 to 0.2)$\times 10^{-3}$ μ^2.

Because of the low density of both the formaldehyde solution (1.07 to 1.1 g/cm^3) and TC-10 (1.16 g/cm^3), the density of the hardened clay drilling mud proved to be somewhat lower than that of the starting mud.

The specimens were found to swell somewhat in fresh water, but suffer practically no changes in dimensions after 30 months' storage in a sodium sulphate solution that is highly corrosive to ordinary cement stone. When kept in a magnesium chloride solution, the specimens suffered a sharp reduction in size during the first two weeks, which could be explained by additional cross-linking of the macromolecules at the OH groups of phenol rings, this being testified by the high strength of the specimens stored in this solution. The strength of specimens stored in distilled water and a sodium sulphate solution was found to remain practically unchanged.

The available data allows one to conclude that the above compositions can successfully be used for isolation work in well intervals containing no polyvalent salt deposits. Recent experiments have shown that the effect of polyvalent cations can be inhibited by introducing into the hardenable clay drilling mud 3 to 5 % of sodium salts of higher fatty acids. When held for 48 hours in boiling saturated solutions of magnesium and calcium chlorides, specimens containing such an additive have shown no changes in linear dimensions, while their strength has been found to increase by 30 to 50 %.

Investigations into the processes of hardening of clay drilling muds with alkylresorcinols and aldehydes have allowed us to reveal the following.

1. The density of the hardenable clay drilling mud (HCM) is equal to or somewhat lower than that of the starting mud, which allows HCM to be pumped practically to any height from the casing shoe.

2. The filter loss of HCM is close to that of the starting clay mud, the filtrate being capable of polycondensation to yield a solid body, which provides for a monolithic bond to form between the plugging composition and the well walls where the filtrate penetrates the wall mud cake.

3. HCM has a high sedimentation stability and suffers no contraction in the course of hardening.

4. The thickening time of HCM can be adjusted by properly selecting the proportions of its constituents.

5. The strength of HCM stone rises with increasing density of the starting clay mud, reaches 5 to 7 MPa in bending for a mud 1.6 to 1.8 g/cm^3 in density.

6. The water and gas permeabilities of HCM stone are close to zero.

7. The sludge of HCM stone is inert to clay muds.

HCM has been field-tested in primary cementing, eliminating casing leaks, and isolating lost circulation zones in more than 70 wells. The tests have shown HCM to be technically and economically effective in eliminating casing leaks and isolating lost circulation zones.

Laboratory Work 11

Determination (qualitative) of water-repelling characteristics of the cement.

I. Level out 5-10 g of cement on the surface of a dry glass and apply a few drops of water to it. On hydrophobic cements water remains in the form of drops that roll freely all over the surface of the cement following inclination of the glass plate. Non-hydrophobic cements avidly absorbs water.

II. Pour cement into a dry glass. Cautiously pour in water along the wall of the glass. In case of hydrophobic cement, water can be decanted in 1-2 hours and the powder will remain almost dry. An ordinary cement will absorb water during this time, forming a paste or a suspension.

Chapter 14

Facilities and Structures for Transporting, Mixing, and Storage of Plugging Materials

Important prerequisites for the preservation of plugging properties of cements are their adequate packing (sacking), proper storage, and transportation. Inadequate storage and transport may cause intermingling of cement from different lots and result in possible breakdowns. Improper storage of cement leads to increased losses and dustiness of storage rooms and creates antisanitary work conditions in loading operations.

Rational organization of transportation and storage of cementing materials, adequate utilization of special structures and mechanical facilities help cut down the cost of cementing operations, improve conditions of work, and raise the production efficiency.

In keeping with the requirements stipulated by the pertinent USSR standard, the size of cement lot shipment is fixed at 200 t. A consignment of cement of a lesser weight is also to be considered a shipment lot. In shipment by water, the size of cement lot may be increased up to 300 t.

When shipping cement in paper sacks, the manufacturing plant must provide each sack with a label pasted on it. On the labels are to be indicated the number of the lot (consignment), the type of cement (for "cold" or "hot" wells) and the date of shipment. It is customary that the quantity of the sacked cement transported in railroad cars be arbitrarily considered to amount to 20.5 t per car.

Types of packing. The plugging cement put out by the Soviet mills reaches the consumers either packed in paper sacks or in bulk. Eeach paper sack holds 48 to 50 kg of cement. The sacks are made of five layers of compact paper, two to three of which being impregnated with a bituminous water-repellent compound. In special cases and when cement is shipped for export, the paper packing is reinforced with an outer cover of sacking.

To keep intact the properties of plugging cement during its long-term storage, effective proves the packing of cement in special reinforced sacks with a cellophane lining or with a cellophane insert. The latter are made by using various cellophane types. When plugging cement is kept in reinforced sacks with cellophane lining, its quality remains unchanged for a long time.

Utilization of the general-purpose transport facilities for the in-bulk hauling of cement requires application of special unloaders. In unloading powder-like materials arriving in bulk in ordinary box-cars, pneumatic suction unloaders Model C-347 and C-362 are employed. Because of their high power consumption, the use of pneumatic suction unloaders is confined to big cement storehouses. Most effective prove unloaders of the blower type, Models C-600Б and C-653H, equipped with a horizontal discharge screw conveyer. These unloaders enable cement to be discharged over a distance of up to 25 m horizontally and to a height of 30 m, this providing for its single-stage reloading from railway cars into the silo banks of all railwayside stores.

Transportation of cement in bulk has a number of advantages over its shipment in sacks. The cost of the usually disposable paper packing alone accounts for over 10 % of the cost of cement. Moreover, the delivery of sacked cement entails labour-consuming reloading operations and high losses of cement due to its dispersion and residual amounts left in the sacks.

Railroad transportation of cement. The method of transporting cement from the manufacturers to reloading centres depends upon the availability of special cement storage facilities and unloading mechanisms at such centres. Where reloading centres are provided with storage facilities for keeping powdered materials in bulk cement can be delivered either in special transport vehicles or in general-purpose ones. Advantages of the haulage by the first method include a reduced scope of labour-consuming loading and unloading operations, elimination of losses due to dispersion in loading, and protection of cement against the harmful effects of atmospheric precipitation. But all special transport vehicles are disadvantageous in that they inevitably have to make dry return runs, and so they are economically profitable only where the distance to be covered does not exceed 300 to 400 km.

Shipment of cement by water. In the USSR, general-purpose barges are used for internal transportation of cement and other powdered materials. As stipulated by the delivery conditions according to the USSR standard GOST 1581-78, a single lot (consignment) of cement should not exceed 300 t, which is mostly much below the load-carrying capacity of a burge. In shipping of sacked cement by barges, there is no danger of different lots of cement being mixed up. Therefore this type of shipment is at present most popular.

As stated before, the transportation of cement in bulk by barges has a number of advantages. But it is admissible only if the following special conditions are observed: (1) strict separation of lots from one another (i. e., shipment of cement by barges having individual compartments of about 300 t capacity each); (2) thorough cleaning of unloading machines before discharging the next lot of cement; (3) providing mooring places with storage facilities for keeping in-bulk cement.

The in-bulk cement transported by standard barges is discharged with the help of a system of mechanical or pneumatic unloaders, either of the suction or blower type. In the USSR, the predominant method of discharging the in-bulk cement from the barges involves the use of floating reloaders equipped with suction-type unloaders.

Technical Data on Cement Carrying Tank Trucks

Model	Manufacturing plant	Truck chassis Model	Load carrying capacity, kg	Empty weight, kg	Number of axles	Number of wheels
C-571	Priluksk plant of construction machines	ЗИЛ-164А	700	2 500	1	4
C-570	Pavshin mechanical plant	МАЗ-504	12 000	3 500	1	4
C-652	Same	КрАЗ-221 of the truck tractor type	24 000	6 550	2	8

The in-bulk shipment of plugging cement by water is justified by the fact that this material finds extensive application in big oil and gas districts.

Transportation of cement by motor vehicles. An efficient way of hauling cement from cement mills and central storehouses to consumers is the use of motor vehicle transport. Of late, very popular have become cement-carrying tank trucks.

Cement-carrying tank trucks with gravity flow discharge. This category of vehicles includes tank trucks wherefrom cement is discharged by gravity on tilting the tanks. Among the trucks of this category, in use in this country are Models ЦС-1 and КАЗ-601 of similar construction. The ЦС-1 Model tank truck is mounted on the ЗИЛ-585 Model dump truck chassis. The tank of this vehicle is of an elliptic section and has a draw (discharge) hole also of an elliptical shape. The cover (lid) of the hole is lifted by an air-cylinder controlled from the driver's cab. In the travelling position, the lid of the hole is tightly held down by means of two screw clamps. Cement is discharged when the front end of the tank goes up, the tank being then tilted at an angle of 48°. Complete discharge of the material is secured by the operation of two pneumatic vibrators set up underneath the tank.

The КАЗ-601 Model cement-carrying tank truck is a modification of the КАЗ-585В Model dump truck and differs from the ЦС-1 Model vehicle only in that it is equipped with a system of levers that opens or closes the discharge hole when the tank is being tilted up or down.

The tank trucks ЦС-1 and КАЗ-601 have a number of shortcomings when employed for delivery of cement to drill rigs (insufficient load-carrying capacity, the need for installing a charging screw conveyer when reloading the material into the cement mixer).

Cement-carrying tank trucks with pneumatic discharge. Unlike the ЦС-1 and КАЗ-601 Model tank trucks, the vehicles with pneumatic discharge can reload cement from the tanks directly

Table 36

with Horizontally Arranged Tanks

Overall dimensions, mm			Tyres (size), mm	Tank diameter, mm	Angle of tank tilt, deg	Number of airslides	Cement delivery height, m	Discharge rate, t/h
length	width	height						
6 100	2 350	2 900	260×20	1 400	7	1	20	20-40
11 250	2 700	3 200	12.00×20	1 600	6	2	20	25-50
13 200	2 700	3 800	12.00×20	1 800	6	2	20	90-70

into storage facilities or into the hoppers of cement mixers without any need for additional unloading mechanisms.

As regards the shape and arrangement of the tanks, vehicles with pneumatic discharge are available in two classes, *viz.*, those with vertically arranged tanks of cylindrical-conic or spherical-conic form and the ones with horizontal tanks of cylindrical shape with spherical end walls.

The advantage of vertical tanks consists in their somewhat higher discharge efficiency and smaller unit consumption of air. Among the merits of horizontal tanks are the smaller metal bulk of their bodies and their better stability on the move, which is due to the fact that their centre of gravity lies lower.

For transportation of in-bulk cement in the USSR are used cement carrying tank trucks with vertical tanks, Models C-386, C-386A, and "Chepel" (of Hungarian make).

Presently, the Soviet industry manufactures several types of cement-carrying tank trucks with horizontally arranged tanks. Widely used are Models C-571, C-570, and C-652.

In oil-fields, cement mixers are often employed for the haulage of cement in bulk. Most suitable for this purpose are cement mixers of the CM-10 and CMП-20 Models, because the capacity of their hoppers is in line with their load-carrying capacity (Table 36).

Sacked cement can be transported in drop-side trucks, provided that the following rules are adhered to: (a) the body of the truck should be thoroughly cleaned; (b) no gaps should be left in the floor of the truck body; (c) cement is to be fully covered with tarpaulin and the ends of the latter should be fastened on the outside of the body panels.

14.1. Plugging Cement Storage Regulations

In storing cement and other plugging materials, the following conditions must be fulfilled: (a) the material must be kept so as to prevent any degradation of its standard quality; (b) no intermingling of cement from different lots is to be allowed; (c) losses in handling are to be minimized; (d) loading must be convenient and safe.

Storage of sacked cement (Fig. 98). Long-term storage of cement is admissible only indoors, in dry rooms. Cements of individual lots should be kept in separate stacks. A passage not less than 2 m wide should be left free between the stacks. The height of each stack must not exceed 10 to 12 sacks placed horizontally. In stacking cement sacks, care is to be taken to see that the tags pasted on the sacks are at their top. During discharging and loading of cement, the sacks often get torn, which results in its spilling and waste. In such cases, the whole of the spilt cement must, on termination of work, be removed from the storehouse, or else kept positively separated. Cement sacks opened to take samples for assaying are to be kept separately from the other sacks.

During its long-term storage, cement should be tested once every ten days. To cut down the waste of cement, samples in each case should better be taken from the same sacks and, for this reason, the opened sacks should be kept till the entire lot has been used up.

During prolonged storage of cement in sheds, the sacks must be restacked once a month, so as to preclude caking (compression) of cement in the sacks.

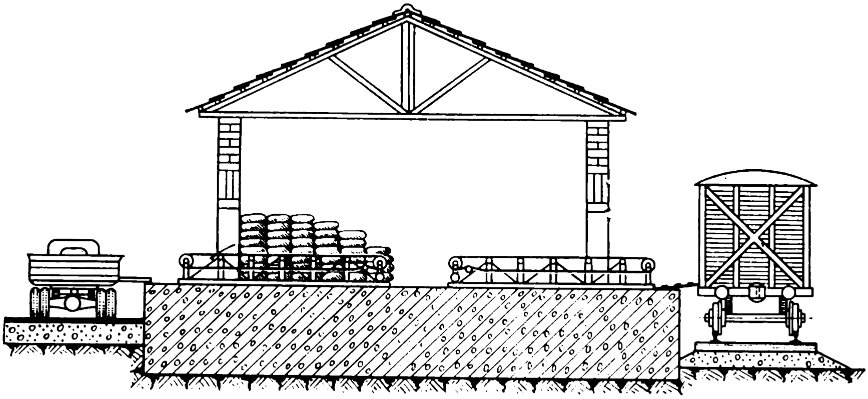


Fig. 98. Sacked cement storage

At drill rigs, cement is to be placed on special platforms (stages) arranged at the sites of anticipated installation of cement mixers. The quantity of cement on each of these platforms, or on specially equipped sledges, must not exceed 20 t, i. e. the hopper capacity of a single cement mixer. The floor of the platform or of the sledge is to lie not less than 50 cm above the ground level. Stacking of cement sacks on the platform should be done in such a way as to create maximal convenience for pulling down the stack.

Prior to stacking, the sacks should be undone or the seam at their butt end cut open. Then, a "wall" having a shape resembling a trihedron prism is to be built up of the sacks, the rest of them being laid down in raws of 4 to 5 sacks each, sloping at an angle of 70 to 80°.

Pulling down of the stack when mixing cement with water or charging the hopper of the cement mixer should be started from the side opposite to that of the "wall".

To protect cement against harmful effects of atmospheric precipitation, the stack is to be covered with tarpaulin, its ends being fastened to the borders of the platform's floor.

In-bulk storage of plugging materials. In storing in-bulk cement and other plugging materials, it is expedient to make use of mechanized railwayside stores. There are known two types of such railwayside stores for keeping plugging cement, *viz.*, of 1000- and 2000-t storage capacity.

The difference between these types consists in their storage capacity and in the number of storage facilities (silos). The 2000-t capacity store has four silos of 500 t capacity each, whereas the 1000-t capacity store counts ten 100-t silos.

When choosing between one or the other store modification in building mechanized railwayside stores, one should take into consideration the following conditions:

1. The capacity of the store should be chosen in such a way as to limit the storage of cement in the silos to no longer than one month, since, in spite of good conditions of storage at the mechanized store, cement, when kept for a longer time, is liable to lose gradually some of its quality. Therefore, in oil districts where consumption of plugging cement does not exceed 1000 t per month, it is advisable to build a store of a smaller capacity.

2. In some oil-producing regions where drilling conditions are difficult, different kinds of cement and plugging compounds are applied.

In the North Caucasus, for instance, up to 10 different types of plugging materials are in use. Conformable to storage regulations, the plugging materials must be kept completely separated from one another. In such conditions, one should build stores with the maximum possible number of silos to provide for simultaneous storage of plugging materials intended for dissimilar purposes.

But it is to be pointed out that blast-furnace slag cements should be protected, whatever the circumstances, against even an insignificant amount of unspecified portland cement finding access to them, since at high temperatures portland cement acts as an accelerator. For this reason, keeping slag cements and portland cements at the same store is inadmissible.

The construction of 2000-t capacity cement stores is advisable in oil-producing regions with large-scale drilling operations, where the latter do not require the use of a great variety of plugging materials.

14.2. Arrangement, Operating Principle, and Technical Data of Railwayside Mechanized Plugging Cement Store

The mechanized store is made up of two sections that can operate independently of each other. Such a separation of the store makes it possible to keep and treat cement of two types (intended for "hot" and "cold" wells, for instance), ruling out their intermingling. If need be, cement can be reloaded from one section into the other. A flow sheet of a mechanized railwayside store is shown in Fig. 99.

The major units of the store include unloading facilities, bucket chain elevators, storage capacities (silos), airslides, and a compressor plant.

The loading and unloading of the mechanized store are effected according to the following principle (Fig. 100). Car *22* with the cement is pushed onto the unloading platform (stage) provided with charging holes *19* (one in each section). The charging holes have adjustable (telescopic) hoses *23* which, with the help of pneumatic jacks *20*, are brought into the working position and are firmly fixed to the discharge openings of the car. When entering the charging hole, cement is entrained by screw conveyer *18* and, via a cleaning arrangement, is fed to the lower end of elevator *14*. The elevator buckets grab the cement, lift it up, and then pour it down into the top airslides. Then cement is directed along the airslides into silos *4*, *5*, *6*, and *7*. The direction of the cement stream into some or other silo is effected by means of slide gates. At the top of the silos, air-bleed holes *3* are provided to release air from the silo as it gets filled with cement. The bottoms of the silos have a layer of special ceramic tiles *11* through which the air, forced by the compressor, is made to pass. The tiles,

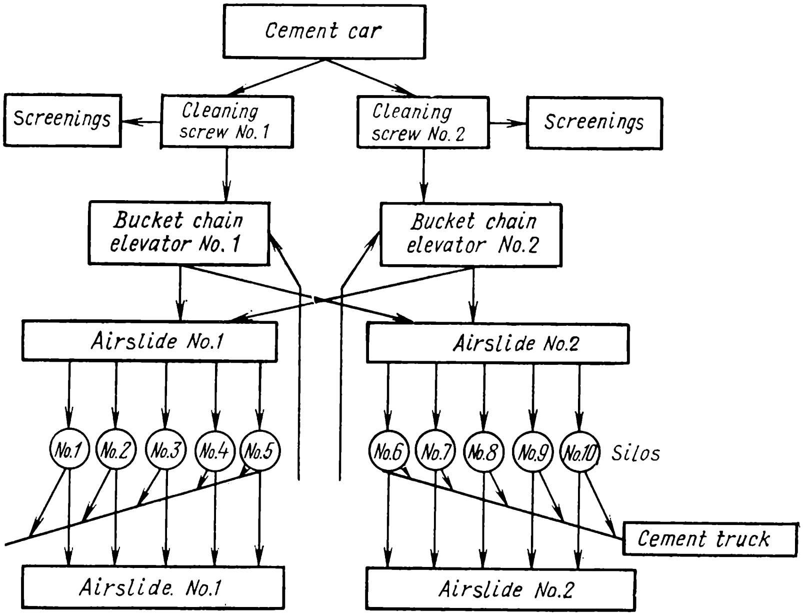


Fig. 99. Flow sheet of a mechanized railwayside store

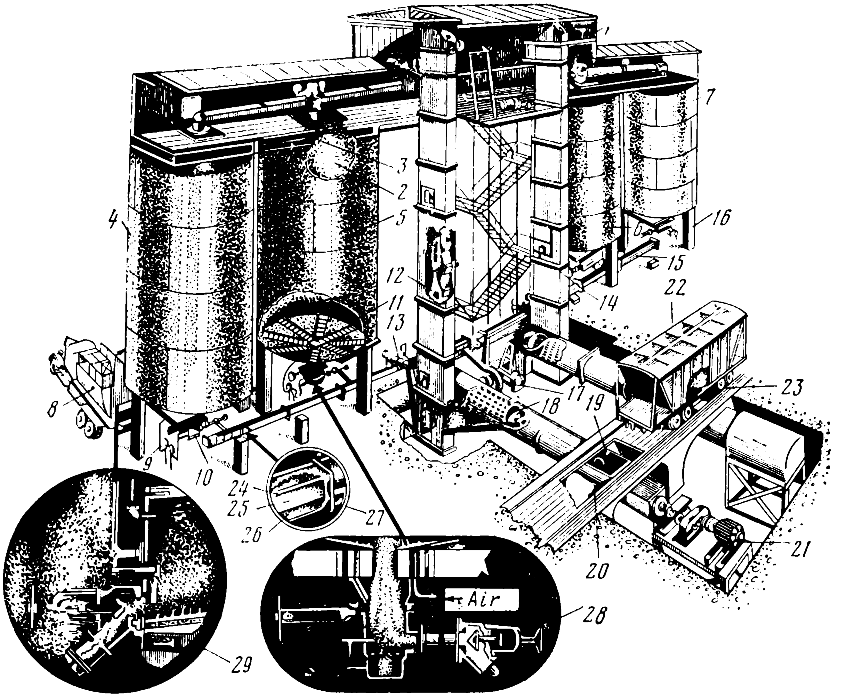


Fig. 100. Mechanized railwayside plugging cement store

1, *17*, and *21* — electric motors; *2* and *24* — cement; *3* — inspection holes; *4* through *7* — silos; *8* — cement mixer; *9* — tappings; *10* — receiving box; *11* — ceramic tiles; *12* and *14* — bucket chain elevators; *13* and *15* — airslides; *16* — posts; *18* — screw conveyer; *19* — charging hole; *20* — pneumatic jack; *22* — cement delivery car; *23* — telescopic hoses; *25* — fabric; *26* — air; *27* — sectional view of airslide; *28* — bottom pneumatic unloaders; *29* — side pneumatic unloaders

$50 \times 250 \times 200$ mm^3 in size, are porous and are encased in boxes accounting for 25 % of the silo bottom area. Aeration of the lower layers of cement is necessary to get the cement discharged from the silos through side or bottom pneumatic unloaders *29* and *28*, respectively. The hoppers of cement mixers and the tanks of cement-carrying tank trucks are filled with cement via the side pneumatic unloaders, which convey cement to lower airslides *15*, along which it is transferred from one silo to another, the lower airslide delivering cement to the lower part of the elevator and the upper ones transferring it from the upper part of the elevator to another silo.

The transfer of cement from one silo into another is done to prevent its compression in cases of long-term storage. The bottoms of the airslides are aerated by air forced in by high-power blowers. The air is forced into pneumatic unloaders from the compressor plant.

The slide gates and the mechanisms engaged in discharging and reloading of cement are controlled from a control board. The store is provided with light and sound signalling systems.

Technical Data on Mechanized Cement Store

Throughput, t/h	250	
Silo capacity, t	500	
Silo dimensions, m:		
diameter	6	
height	15.5	
Screw-conveyers with cleaning arrangement:		
delivery, t/h	125	
diameter, mm	500	
screw thread pitch, mm	360	
length, m	6.7	
electric motor:		
type	АО-6Э-6	
power, kW	10	
speed, rpm	980	
reduction gear:		
type	Р-10	
gear ratio	19.81	
Elevators:		
delivery, t/h	125	
height, m	26.19	
electric motor:		
type	АО73-6	
power, kW	20	
speed, rpm	980	
reduction gear:		
type	РГП-250×400 mm	
gear ratio	24.4	
Airslides:	Upper	Lower
delivery, t/h	250	50
total length, m	36.8	29
height, mm	360	310
width, mm	400	150
slope, deg	7	4
required air pressure, Pa	2770	1680
air consumption, m³/h	2400	600
air blower:		
type	ВВД-8	
speed, rpm	1350	1250
pressure, Pa	3500	2500
electric motor:		
type	АО51-4	АО41-4
power, kW	4.5	1.7
speed, rpm	1440	1420
Aerated bottoms:		
air pressure, MPa	0.2	
air consumption, m³/min	5	
Bottom pneumatic unloaders:		
delivery, t/h	45 to 150	
discharge hole diameter, mm	100	
air consumption, m³/t	1	
air pressure, MPa	0.2 to 0.3	

The service staff of the mechanized store counts two to four operators per shift.

14.3. GROZNEFT Plant for Preparing Dry Plugging Mixtures

Bank-run sand (Fig. 101) is fed by a lift truck onto a vibrating screen 20 to 25 mm in mesh size. The sifted sand is fed by a belt conveyer into a rotary drum drier where it is gas-flame dried usually at a temperature of 100-120 °C. From the rotary drum drier the dry sand is delivered by a bucket chain elevator onto a belt conveyer and further on into a bunker put up above hoppers. The bunker is provided with a screen 1 to 1.5 mm in mesh size.

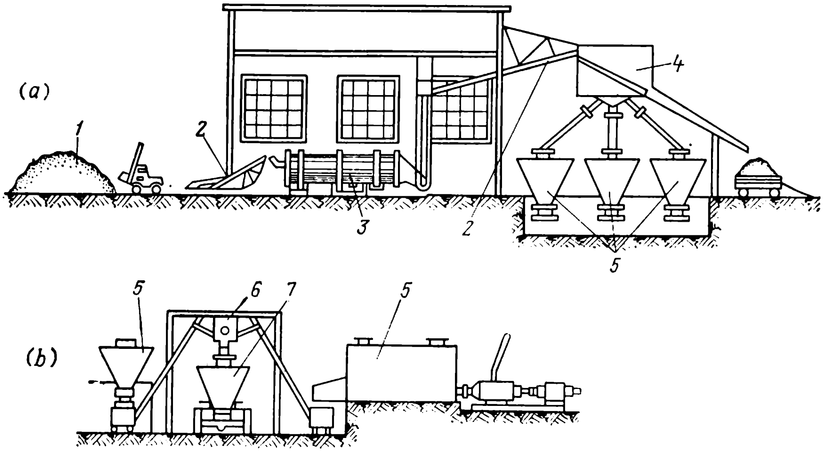


Fig. 101. GROZNEFT plant for preparing dry plugging mixtures

(*a*) schematic diagram of bank-run sand processing; (*b*) schematic diagram of dry mixture preparation and loading; *1* — sand; *2* — belt conveyer; *3* — rotary drum drier; *4* — bunker; *5* — hoppers; *6* — mixing chamber; *7* — 2CMH-20 Model cement mixer

The screenings are diverted along a sloping chute. The tank has three pipelines whose ends are let down into the charging holes of the hoppers. Each pipeline is equipped with a slide gate by opening of which sand can be discharged from the bunker into one of the hoppers. The latter are intended for storage and batching of aggregates (sand, etc.) during preparation or dry plugging mixtures.

Cement is supplied to the installation by the 2CMH-20 Model cement mixers. A screw conveyer-type loader is placed under the discharge arrangement of the cement mixer to provide for charging a bin designed to proportion cement during preparation of dry plugging mixtures. The worm feeders of the hoppers are driven by electric motors via gearboxes. The latter make it possible to vary the speed of the worm feeders, i. e., to ensure the needed proportioning of the binder and the aggregate.

The batching of the binder and the aggregate is determined by the ratio chosen (1 : 1, 2 : 1, etc.).

Dry plugging mixtures are prepared in the following sequence: the inert material (aggregate) is fed from a hopper into the receiving chamber of a mobile rail-mounted screw conveyer-type loader. This loader delivers the material into a receiving chamber fixed at some height. From the bin cement is fed into the same chamber by the screw conveyer-type loader. Cement and sand are grabbed from the receiving chamber by a screw conveyer-mixer which secures intermingling of sand and cement during their transport from the receiving chamber to the discharge opening. From the screw conveyer-mixer the dry mixture is charged into the hopper of the cement mixer, wherefrom it goes directly to the site of the cementing jobs.

In mixing three and more components, when preparing cement-sand-bentonite slurries, for example, it is the bicomponent mixture that is prepared first, which is then mingled with the next additional material.

14.4. KRASNODARNEFTEGAS Plant for Preparing Plugging Mixtures

The plant (Fig. 102) intended for mechanized mixing up of sand and cement, has been constructed at a mechanized cement store.

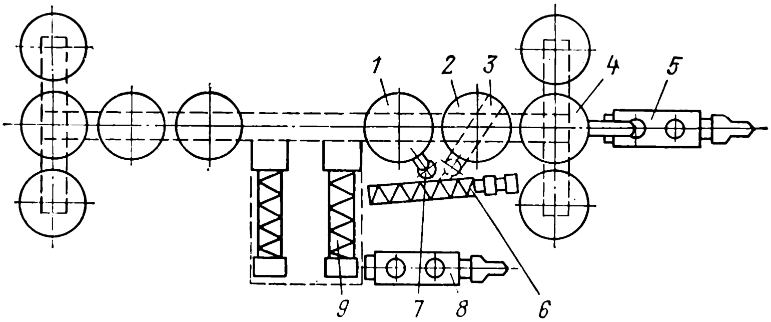


Fig. 102. Schematic diagram of the KRASNODARNEFTEGAZ plant for preparing dry plugging mixtures

1 — sand silo; *2* — cement silo; *3* — feeding screw conveyer; *4* — dry plugging mixture silo; *5* — cement mixer being loaded with dry plugging mixture; *6* — mixing screw; *7* — rotary table feeder; *8* — cement mixer delivering sand; *9* — loading screw conveyer

One of the available cement storage silos *1* is utilized to hold dried quartz sand brought to the store in the CMH-10 Model cement mixers. In the course of mixing, cement is fed by screw conveyer *3* with a continuous delivery of 260 kg/min.

The sand is conveyed by rotary-table feeder *7*. By adjusting the delivery of sand (by means of the rotary-table feeder) a mixture of cement with sand in the ratio 1 : 1, 2 : 1, 3 : 1, etc., can be obtained. The compound is mixed up by mixing screw 6, which delivers it into silo *4* for storage of the ready-made mixture.

In use is also a mechanized method of preparing a cement-bentonite mixture (Fig. 103). Cement mixture *3* is uniformly charged with cement (or slag) and clay. Cement charged into hopper *1* at a mechanized store is then discharged into a special chamber of vertical screw conveyer *2* and, at the same time, mud powder is poured by hand into it in an amount that depends upon the specified composition of the cement bentonite mixture.

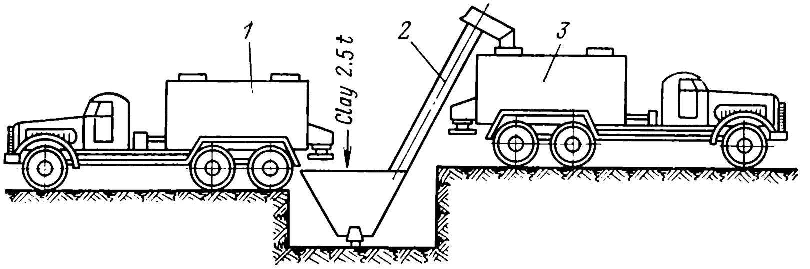


Fig. 103. Illustrating the preparation of cement-bentonite mixtures

Sometimes, concurrent charging of the cement mixer with cement and clay, with subsequent rebunkering of the mixture by using an intermediate hopper, is practiced.

14.5. Laboratory Control over Plugging Materials

According to the USSR standard GOST 1581-78, a sample weighing 10 kg (1 kg from every 10 sacks) is taken from each cement lot (consignment) for assaying. When cement is shipped in bulk by motor vehicles, the procedure of sampling is established by mutual consent between the supplier and consumer. Samples taken from each lot of cement are thoroughly mixed together and halved. One half is marked and kept in a dry, tightly closed container for a period of two months in case of need for re-examination. The second half is tested in accordance with the standard requirements.

The cement samples collected for testing should be sent to the laboratory in an air-tight, if possible, hermetically sealed container. The kind and the condition of the latter is indicated in the log book.

Before testing, each sample is screened and then thoroughly agitated. The sieve residue is weighed and then rejected.

Prior to testing, the cement, aggregates, and water should be kept indoors till their temperature reaches that of the room, which is to be 20 ± 2 °C. The indoor temperature is registered in the log book thrice a day. The vessel used for weighing or measuring of water should be wetted before gauging.

The cement and aggregates are to be weighed to an accuracy of up to 1 g, water to an accuracy of up to 0.5 g, or 0.5 ml, and reagents to an accuracy of up to 0.005 g, or 0.005 ml.

No aluminium and zinc vessels should be used in testing.

I. Training and Briefing

1. Eeach one of the new workers engaged in the laboratory is to be briefed on matters concerned with safety engineering and industrial sanitation (hygiene).

General briefing is done by the safety engineering official of the organization (institute).

During general briefing the worker is to be familiarized with safety regulations in dealing with electric appliances, with fire safety rules, with accident prevention in operating vessels working under pressure, with rules governing handling of chemical agents. The worker should also be familiar with the plan for evacuation from the premises of the organization (institute) in case of fire and know ways of rendering first aid to victims sustaining electric and other injuries.

At the workplace, the worker should be acquainted with the rules for safe attendance to the equipment, with diagrams, circuits, documentation.

2. The newly admitted member of the staff is handed the present instruction for study and guidance.

3. After the introductory briefing at the workplace, and after studying by the new staff member of the present instruction and mastering by this member of safe operations in performing the job, the practical knowledge of safety regulations and of safe conduct of work are put to the test of examination following which the worker is allowed to perform the assigned tasks.

The acquired knowledge is put to the test of examination by a commission composed of the head of the laboratory or of the group, safety engineer and a representative of the trade union.

4. Repeated briefing on safety engineering is done by the head of the department, group or laboratory. It covers all the workers and is to be practised at least once a year.

5. After secondary briefing the knowledge of safety regulations is again to be put to the test of examination.

6. The results of briefing and examinations are recorded in a special card of briefings on safety engineering.

Persons falling to pass the examination are not allowed to do the work on their own.

7. The executives are examined for their knowledge of safety regulations by a commission headed by the chief engineer of the organization (institute) at least once every 3 years.

II. General Safety Engineering Rules

8. Before commencing work, the workplace should be properly prepared, *viz.*, all superfluous objects removed, the necessary tools and appliances have to be checked and be in good order and repair.

9. Before getting down to work, one should become acquainted with the procedure to be followed in doing the assigned job, with technical conditions, features peculiar to performing this or that operation, and also inspect the condition of the equipment and apparatus, their working order, the availability of all the necessary and well-functioning interlocking devices, of shut-off accessories, control and measuring instruments, which are to be checked for their proper operation.

10. When doing work, protective garments (if these are provided for by the standards) should be worn.

11. It is prohibited to perform non-assigned tasks, exception being made for those related to prevention and elimination of breakdowns, fires, accidents, etc.

12. It is prohibited to leave unattended vessels operating under pressure, switched on electric heaters and chemical agents made ready for use.

13. Wrenches should be in good order and of adequate sizes. No inserts between the wrench jaws to change the size of the wrench are allowed, nor is it permitted to use pipe pieces, crow-bars and other objects intended to extend the handle of the wrench.

14. Files and screw-drivers should be provided with wooden handles and rings fitted on their ends.

The handles of hammers should be firmly held in place through wedging. All wooden handles of the fitter's hand tools must be made of durable non-breakable wood with no cracks.

The business (striking) end of hammers, chisels, etc., should undergo treatment in good time, to remove burrs, cold hardening.

15. Once the work has been terminated, it is necessary to see to it that all the pieces of equipment and apparatus are switched off, to tidy up the workplace, to clean it from the dirt and see to it that all the chemicals are stowed away.

III. Vessels Operating under Pressure

16. All the autoclaves operating under pressures higher than 100 MPa should be installed in special cabins. Persons who are not directly engaged in operating the autoclaves are to be prohi-

bited the access to these cabins. No cabins are envisaged for the autoclaves operating under pressures below 100 MPa.

17. During operation of the autoclaves, signs reading "Attention! Autoclaves under pressure" are to be posted up on the cabin doors.

18. The autoclaves, hydraulic presses and other vessels operating under pressure must be fitted out with safety valves.

19. Pressure gauges should carry a seal or a stamp of the state inspector and be tested at least once every 6 months.

20. The pressure gauges are provided with three-way cocks which are mounted so that on turning off the cock for blowing, the jet of the liquid or gas can be directed away from the people and equipment.

The blowing is done with the pressure in the vessel not exceeding 1 MPa.

21. Pressure gauges are selected so that the working pressure should cover approximately 2/3 of the gauge dial (starting from the zero point).

22. A red line is drawn on the pressure gauge dial indicating the maximum permissible pressure in the given vessel. This red line should not be shifted arbitrarily during operation of the gauge.

23. Pressure gauges with a broken glass, loose dial or body, overdue inspection, or without the seal and stamp of the state inspector, with the pointer failing to return to the dial zero in the absence of pressure are considered to be in the state of disrepair and their use is to be discontinued.

24. All the vessels operating under pressure have to be provided with locking (shut-off) accessories designed to withstand the pertinent pressure, as well as with safety valves.

25. All the vessels operating under pressure, their fittings and pipelines should be tested after being assembled under a pressure exceeding the working one by 25 %. Periodical tests under the same pressure are to be made every 3 years, this being followed by a defectoscopic investigation of all the subassemblies and components of the equipment.

26. The autoclave and consistometer units must have control block-diagrams indicating all the distribution and supply lines, shut-off accessories, pipelines, control and measuring instruments, etc.

27. Cocks, valves, change-over switches and starting devices should be provided with inscriptions as to their purpose.

28. All the pipelines operating under pressure must be laid down in special troughs or protected by deck boards or housings.

29. All the pins, shims (spacers) and other fastenings of the autoclave covers should be inspected every time before the autoclaves are put in operation. In the face of a slightest fault

in any of the components, the operation of the autoclave is to be barred.

30. Safety valves are inspected once a month, with the results of the testing recorded in a special log book.

31. Safety valves should be adjusted for a pressure exceeding the working one by not more than 10 %.

32. All the vessels operating under pressure must have certificates attached to them carrying records of inspection, the names of persons responsible for their failure-free operation and an indication as to the place of installation. In addition to this, a log book is to be available in which are made recordings of the results of testing of return (check) valves, repairs of the equipment, and replacement of individual components and assemblies.

33. The heating of the autoclave and the growth of pressure therein should proceed under a continuous control of the operator. In case of the pressure exceeding the preassigned level, it is to be reduced by half-opening the escape (blow) valve.

Pressure reduction by manipulating the safety valve is not allowed.

34. In the course of the autoclave operation, the valve between the hydraulic press and the autoclave should be kept permanently closed.

35. The autoclave cover is to be kept firmly fixed in place. Not a single pin is to be left unfastened.

IV. Chemical Reagents

36. All the operations associated with the evolution of gases harmful for the organism are to be done under vent hoods provided with an exhaust ventilation system.

37. Virulent poisonous substances should be kept in a storeroom. Their keeping in the laboratory itself is to be prohibited.

38. Transportation of flasks chemicals is to be allowed only in specially fitted carriages and when carried by hand, on special borrows.

39. When pouring acids from the flask, an appliance for gradual inclination of the flask is to be used.

40. When pouring an acid from the flask into other vessels, a special headpiece that prevents splashing of the acid is to be fitted on the vessel's neck.

41. The use of a siphon is allowed in pouring acids. A special appliance should be employed in sucking the acid into the siphon.

42. In making acid mixtures it is the sulphuric acid that should be last to be poured in.

43. When preparing a solution, it should be an undeluted acid that is to be poured into water not vice versa.

44. Nitric acid should not be allowed to come into contact with rags, wood shavings, saw-dust, etc.

45. Neutralizing solutions have to be kept constantly ready in places where acids and alkali are dealt with. In case of an acid or alkali contracting the skin of an operator, the affected place should be washed right away with a neutralizing solution and then with a soaped water.

46. With a potassium bichromate solution touching the body, the affected place should be dabbed with a 5 % hyposulphite solution.

47. As an acid neutralizer, a 10 % sodium bicarbonate solution is employed. Alkalies are neutralized by a 10 % acetic acid solution.

48. In the event of second degree burns followed by the development of blisters, these should not be either opened up or punctured. A dressing made of sterile material is to be applied to the burned skin and the victim should be immediately sent to the nearest medical post.

49. In case of vertigo or nausea developing in an operator, he should be urgently suspended from work untill the physician allows him to continue it.

50. In a premise containing apperatus carrying mercury, the air is to be assayed for the mersury fumes at least twice a year (and invariably once in summer time).

51. The stock of mercury must be kept in a storeroom in thick-walled glass-ware with ground-in stoppers or in sealed iron containers. The vessels should be placed on griddles.

52. Glass containers for mercury should have a capacity of not more than 500 cm^3 each.

53. To get mercury from the storeroom a permit issued by the laboratory head is required, and its amount should not exceed that needed for a one-time use.

54. The mercury apparatus is to be placed on an enamelled or an oil painted griddle.

55. Every time when mercury is spilled accidentally it should be immediately collected. To prevent mercury getting rubbed into the floor and spreading all over the premise, the gathering of the mercury drops is to be started from then periphery of the defiled area of the floor. Mercury is collected by using tin plates or tin-plate brushes, the amalgamation being achieved through rubbing mercury onto them. The mercury drops that adhered to the amalgamated surface are to be shaken off into a vessel with water, closed with a rubber plug.

The amalgamating plates and brushes are to be kept in a vessel plugged with a rubber stopper.

56. Should any mercury drops find their way in between the flaps of the laboratory table, the flaps must then be detached and mercury collected.

57. After collecting the spilt mercury, the floor and the equipment should be thoroughly washed by a weak manganese solution, and after this the laboratory air is to be assayed for the presence of mercury fumes.

V. Electrical Equipment

58. All the current-carrying parts should be insulated and protected against any casual contact with them.

59. All the electrical installations must be grounded in accordance with the "Rules of technical exploitation and safe servicing of electric installations at industrial enterprises". Subject to grounding are metallic parts of electric equipment and installations that may become energized because of faulty insulation.

Grounding of electric installations is not obligatory: with a rated a. c. voltage of 380 V and below in dry premises with dry poorly conducting floors (wooden, asphalted, etc.), if these electric installations are protected against simultaneous contact with them and some other grounded objects;

with a rated a. c. mains voltage of 127 V in all premises, except for explosion-hazardous and outside installations.

60. Starting devices of the electric equipment set up in the open (outdoors) and in damp premises and in those with cement floors should be provided with dielectric rugs or insulating supports.

61. All the insulating objects should undergo testing:

dielectric gloves, at least once in 6 months;

insulating rugs, not less than once in 2 years;

insulating supports are inspected not less than once in 6 months.

62. The insulating rugs and dielectric gloves are to be kept in a specially allotted place.

63. Eeach starting device should carry an inscription as to its purpose.

64. Electric receptacles should have inscriptions indicating the mains voltage.

65. The construction of plug-receptacles intended for different voltages must differ from one another so as to rule out any possible connection of electric equipment or electric tools to the mains of wrong voltage.

66. All the wiring panels, electric knife-switch housings and other starting devices should be kept under lock and key.

Persons other than electricians are prohibited to tamper with them, to replace cartridge fuses and also make repairs.

67. Distinctive warning signs "Lightning" should be provided on the front side of all doors, covers and housings enclosing current-carrying parts.

68. Starting devices must be set up in readily accessible places with free approach to them.

69. The start button of push-on starters must be of a different colour than the stop button and should lie flush with the block.

The stop button must be of red colour and project from the block by 2 to 3 mm.

70. Cartridge fuses should match the magnitude of the mains current. Insertion of jumpered fuses in lieu of calibrated ones is prohibited.

71. Autoclaves fitted out with electric heating plates having swinging open covers should be provided with interlocking safety devices that cut off the electric current supply to the coil of the plate when this is laid open.

72. Prior to opening the heating plate in order to cool down the autoclave, the electric power is to be switched off by means of a special cut-off device. To use the interlocking device for this purpose is prohibited.

73. The wire connecting electric installations to the mains should be protected against any mechanical damage along its entire length over a height of 1.5 m from the floor lever.

74. Inside lighting of the vent hood is to be effected by explosion-proof luminaires or by standard ones set up outside of the hood (through glazed windows).

75. Outside luminaires should be enclosed in glass globes.

76. Electric wiring in vent hoods must be protected against the action of chemicals.

77. No plug receptacles should be admitted inside the vent hood.

78. Eearthing arrangements have to be tested twice a year and once invariably during the driest month of the year.

VI. Industrial Sanitation (Hygiene)

79. Each laboratory room is to be provided with a medicine chest complete with all the needed medicaments.

80. Autoclaves and vent hoods must have exhaust ventilation.

81. Tables intended for handling chemical agents must be faced with stainless steel and wall panels nearby these tables should have a glazed tile facing.

82. Laboratory floors must be covered with linoleum.

83. Water sinks must have a glazed tile wall facing all around the water-running cock.

84. The effluent pipeline from the autoclaves should be led out from tbe laboratory rooms into a special pit provided with a cover.

85. Chemical reagents must not be disposed into the general sewer system without being preliminarily neutralized.

86. Sludge and slime must not be disposed into the sewerage. A special pit is to be arranged for this purpose outside the laboratory premises.

87. Artificial illumination at workplaces is to be not inferior to 200 lx with fluorescent lamps, and not less than 100 lx with incandescent ones.

88. At the sites of the ball mills the artificial lighting should be not inferior to 20 lx with incandescent lamps.

89. Ball mills must be put up outside the laboratory premises.

Chapter 15

Organization of Cementing Jobs. Complications and Safety Engineering in Cementing of Wells

15.1. Organization of Cementing Jobs

To secure failure-free cementing of wells, one should pay serious attention to the proper ogranization of this process.

The drilling office places in advance an order for cementing a well to the cementing office or the cementing department. Depending upon the existing conditions and the nature of the jobs, the cementing units arrive at the drilling rig within a period of a few hours. If cement is charged into the cement mixers at the drilling rig the timing of the arrival of the units coincides with the termination of connecting the machines with the wellhead. But if the cement mixers are charged at a store, then they arrive at the drill rig together with the cementing units.

The cementing units and cement mixers are positioned at the site of the well and are connected with the wellhead in accordance with a pattern worked out in advance.

The cementing units are set up with their measuring tanks as near as possible to the wellhead. The units should be positioned at the site at a distance of approximately 1 m from one another, so as to leave a free passage between them. One cement mixer is placed between two units. From this the ready-made cement slurry is fed to a receiving measuring tank, wherefrom it is pumped into the well by the cementing units.

Once the cementing units and the cement mixers have been put in place, an intake line is laid down, following which the units begin receiving water for mixing it with either cement or a plugging compound. Thereupon pipes of the discharge line are laid down. The units' intake line should be arranged to serve also the purpose of receiving the fluid for displacing the cement slurry.

The connections with the cementing head put up at the wellhead are made by means of an all-metal flexible hose. With a large number of cementing units, several machines can be hooked up to form a single line.

Effective patterns for arrangement of cementing units and cement mixers have been elaborated in different oil and gas regions of this country, which permit cutting down in a large measure the time and the cost of cementing jobs and also to lighten the work of the operators. Figures 104*a* and *b* present standard cementing equipment hookup diagrams. Their main difference consists in the number of the cementing inits and cement mixers employed. The given patterns satisfy in principle the requirements for cementing jobs when use is made of cement, cement-sand and slag-sand mixtures. In the event of lightened plugging mixtures, the number of units is steeply increased because of the need for greater quantities of water, particularly in the regions where the demand for it cannot be met over a short period of time.

When a large number of cementing units are engaged in the job, wellhead equipment (АУ-5 Model carriage) is used. Figure 104*c* illustrates a hookup of a wellhead, cementing units and cement mixers machines in performing cementing jobs with the help of АУ-5 Model wellhead equipment carriage. The use of the wellhead equipment reduces the impact loads during pumping of the slurries into the tubing, helps establishing convenient connections of the lines, and cuts down the number of pipes to be tied directly at the cement head.

Figure 105 offers a diagram showing the disposition of equipment in doing cementing jobs with a lightened slurry in No. 1 Galyugaevskaya well which is 5500 m deep with bottom-hole temperature of about 200 °C.

Once the pipes are tied on, the discharge lines are tested under a pressure exceeding 1.5 times the working one. The operations are performed under the supervision of the engineer in charge of cementing.

Thereupon, the executive in charge of the cementing jobs instructs the operators, estimates and proportions the volumes of the slurries that have to be forced into the well, indicates the sequence in wich the machines are to be put into operation, outlines the mode of operations, fixes the working pressure, sets apart the units designed to complete the cementing of the well, and so on.

On termination of the preparatory work and after getting the machines ready for the job, one of the outfits is employed to restore circulation in the well, following which the crew starts with mixing of the cement compound with water for delivery into the well. To do this the hydraulic compartment of the pump and the inlet header are preliminary filled with water. In order to prepare the cement slurry, the operator of the mixing machine opens the barrel of the inlet hopper and when the pressure in the compensator reaches the pre-assigned level he gives a signal to start the batching-feed worm screws. The cementing units then proceed with the pumping into the well of the cement slurry.

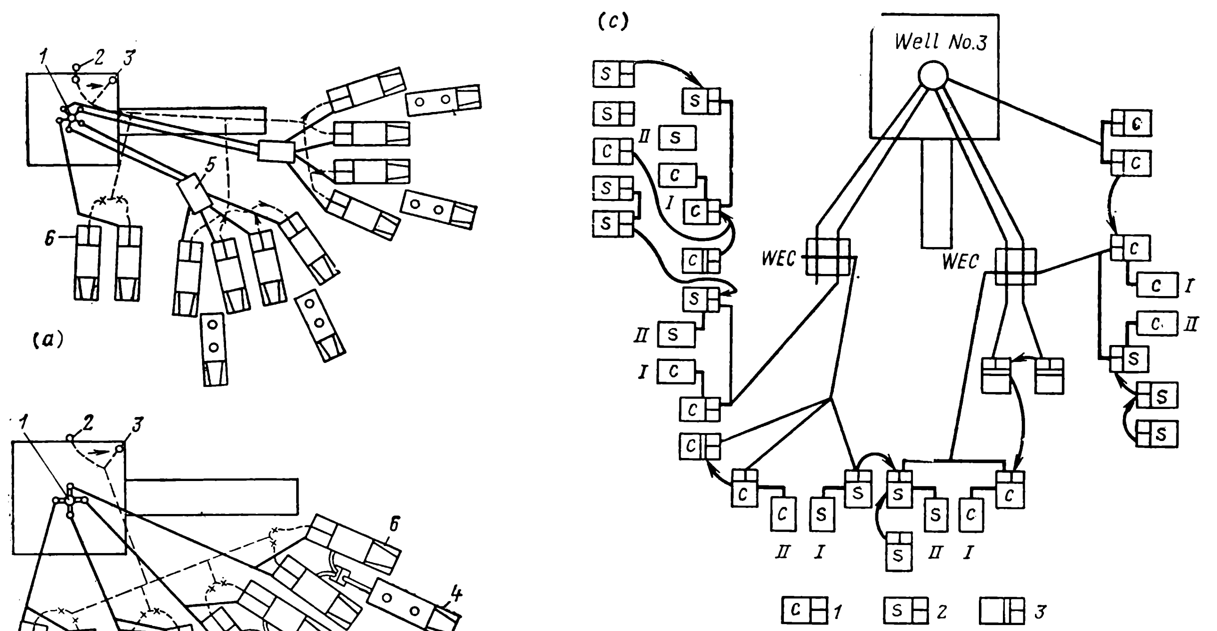


Fig. 104. Typical cementing equipment hookup diagrams

(*a*) with 10 cementing units in use; (*b*) with 8 cementing units in use: *1* — water standpipe; *2* — standpipe; *3* — cementing head; *4* — cement mixers; *5* — АУ-5 Model wellhead equipment carriages; *6* — cementing units; the arrows indicate subsequent transfer of pipelines; (*b*) with two АУ-5 Model wellhead equipment carriages in use: *I* — first mixing stage; *II* — second mixing stage; *1* — cementing unit carrying reagents for cement-bentonite slurry; *2* — cementing unit carrying reagents for slag-bentonite slurry; *3* — cementing unit

In the absence of a cementing process control station, the density of the slurry pumped into the well is measured manually. The composition and properties of the plugging mixture should match the ones recommended at the laboratory.

Once the pumping of the cement mixture has been completed, the discharge lines and the pumps of the cementing outfits are flushed to remove the remnants of the slurry. Having unscrewed the stop bolts of the cementing head, the separating plug is pushed forward and then the slurry is displaced.

With a steep rise of the pressure, the pumping procedure is stopped. On establishing and eliminating the cause responsible for this rise, the operation is re-started only at the word of command given by the superintendent in charge of the operations. The operators count the volume of the displacing fluid pumped in. When the separating plug gets seated on the cement baffle collar, the pressure goes up.

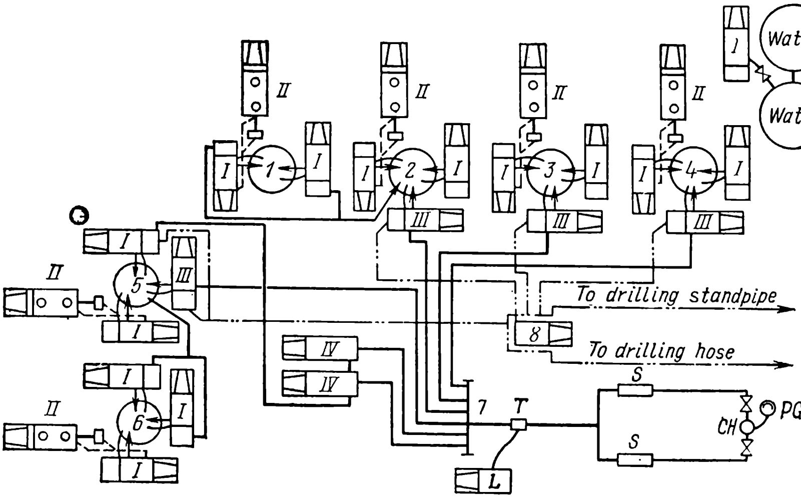


Fig. 105. Arrangement of equipment in cementing No. 1 Galyugaevskaya well with lightened slurry

1 through *6* — slag-sand slurry tanks; *7* — delivery manifold; *8* — drilling mud manifold unit; *I* — ЦА-320М Model cementing units; *II* — СМН-20 Model cement mixers; *III* — 3ЦА-400 Model cementing units; *IV* — АН-500 Model units; CH — cementing head; *T* — flow rate and pressure transducer; *L* — laboratory; *S* — screen; *PG* — pressure gauge

This instant is considered to mark the end of the well cementing. The landing of the separating plug onto the cement baffle collar is effected by one or two cementing units operating in the first gear.

After squeezing in of the clay mud, commonly used as a displacing fluid, the proper sealing of the casing float valve is checked. Then the pipes of the intake and discharge lines are taken apart.

The remaining clay mud is pumped into the receiving tanks of the drill rig, the batchers are flushed to remove the rest of the cement mixture and clay mud, the tools and pieces of equipment are placed on the platforms and the machines are made ready to move on.

The well cementing procedures, the quality of cement, the parameters of the plugging mixture and its composition are recorded in pertinent documents — in plugging operations check records and in a control list. These are indispensable documents in performing all operations involved in cementing jobs.

15.2. Complications in Cementing of Wells

Successful completion of cementing jobs in wells is determined by geological and technical factors. The degree of the influence exercised by these factors, their intensity and the nature of their manifestations vary greatly even within one and the same oil field. Therefore, cementing jobs should be effected, wherever possible, on the grounds of a thorough analysis of the information obtained from preceding geological-geophysical, technical, and running examinations of the wells.

Complications Associated with Pretreatment of the Well Bore

With substandard and untimely working of the well bore, or in the absence of this operation, the walls of the well often develop landing spots, and in the event of a poor quality mud fluid, a slimy clay coating (mud cake) is deposited on them.

A narrowing of the well bore can prevent the string from reaching the bottom of the hole, so that the string may get stuck in the well.

The following causes generally account for jamming of casing strings: narrowing of the well bore, soiling of the latter owing to insufficient flushing, adhesion of the casing, hydrostatic pressure of the flushing fluid, and caving of the rocks.

The sticking of the casing due to adhesion (under the effect of hydrostatic pressure) occurs in cases where the string is left immobile in the well. To prevent this, the casing string should not be left immobile, and the flushing fluid must carry additions of various substances displaying "lubricating properties".

The main causative factor responsible for the sticking of casing strings is inadequate cleaning of the well bore from the drilled-out rock due to the reduction in the amount of pumped-in flushing fluid with increasing depth of the well.

To avert complications stemming from the narrowing of the bore, the latter should be thoroughly worked before the casing is run down. It is advisable that the rate of the slurry movement should be kept at more than 1 m/s, the viscosity of the clay mud should not exceed 50 s, and its gel strength should not be higher than

13 to 17 Pa in 10 minutes. In many cases, it is expedient and even necessary to gauge (profile) the well bore with rigid tools.

A wrong estimation of the volume of the caverns may result in the lifting of cement slurry either below or above the mark. In the first case, several horizons will be left unsealed and in the second, the pressure during pumping of the slurry will be increased.

To provide for a uniform distribution of cement slurry, the casing string is to be properly centered.

Pretreatment of Plugging Mixtures, Their Preparation, and Peculiarities of Application

The cementing of uncomplicated and relatively shallow wells is effected through mixing of pure plugging cement with water preliminarily untreated with chemical agents. But at elevated bottom-hole temperatures and pressures, the same cementing process requires an extremely strict observance of all conditions set up by the pertinent oil-field laboratory or the branch research institute laboratory.

One of the capital points is matching the composition of aggregates, active additives, and chemical agents, used in large quantities in the field, with the formulations of mixtures elaborated (selected) at the laboratory.

Increasing depth, rising temperatures and pressures, lack of special cements make it necessary to employ plugging mixtures of complex compositions. However, one should always strive for preparation of the simplest compounds, convenient in use.

As concerns the setting time (initial and final) of their slurries, cementing materials are presently chosen with reference to the pressure actually built up by the flushing fluid column and to the dynamic or static temperatures of rocks at the bottom-hole of wells.

When doing cementing jobs, the materials should be chosen in conformity with concrete conditions prevailing in each individual well. Each lot of cementing materials intended for casing a given well is to be put to the test under actual bottom-hole conditions. The choice is to be made of the recommended compounds whose composition and materials are to be in actual use at a concrete well.

Stale cement must be screened to remove therefrom compressed (caked) cement lumps prior to its dispatching to the drill rig or before charging in cement mixers. Cement contaminated with various extraneous objects (gravel, fragments of boards, paper, etc.) should be invariably sieved. When using cementing materials mixed with sand (quartz or hematite), clay, opoka, etc., they should be thoroughly intermingled.

In preparing a compound, one should not use sand, clay, or opoka of elevated humidity (approximately more than 3 %), so as to prevent the formation of lumps in bins (hoppers), the presence of which in the mixture of binders can put out of action cement mixers and frustrate the cementing operation.

It is common knowledge that the longer the cement is left in bins (especially during the spring, autumn, and winter months), the more compressed it becomes, and when it is fed with screw conveyers, the latter are liable to get out of action, (keys get shorn off, hopper motors run under heavy loads, etc.).

When a compressed cement flour is being discharged from cement mixers, there develop caving domes therein, which hamper the conduct of the operations: the formed cones have to be brought down and the density of cement slurry must be adjusted, for a nonuniform feeding of cement tends to create abrupt fluctuations in the density of the slurry. The "bridging" of cement mixes is most often observed when these carry moist, water-absorbing additions of the bentonite clay type and, especially, of swallen perlite.

Upon introduction of large amounts of the CMC (1 % and more) into cement slurries (and particularly cement bentonite), it seems as if they get set prematurely. But with their further stirring, the viscosity declines and the slurry becomes thinner though not always to a sifficient degree.

As the conditions at the drill rig are sometimes not favourable (especially in winter time) for dissolution of the CMC, the reagent is poured into the receiving tanks of the units and then "broken up" by means of a water jet delivered under pressure by the pump of the outfit in order to speed up the dissolution procedure. But the retarder (particularly when in a large amount) fails to dissolve completely. In mixing cement with water carrying an incompletely and non-uniformly dissolved CMC, the consistency and the viscosity of the resulting slurry vary over a broad range and this yields batches of slurry that are poorly pumped or brings about caking of the slurry in the mixing chamber of the bunker. When going to the receiving tank, the slurry is fed in intermittent batches with over- and underflow.

While preparing cement-bentonite slurries, one must keep an eye on the supply of the mix into the bin and bring down the piled cones that develop therein. The bringing down of the material can be followed by its compression in the mixing chamber, an increased viscosity of the slurry and a rise of the pumping pressure.

The use of large amounts of SAL (approximately 0.4 to 0.5 %) as a retarder is conducive to (a) foaming that impairs the conditions of cementing and causes precipitation of sand and the weighting agent in the receiving tank in case when weighted or sand compounds are employed; in some instances the quantity of foam is so great and that of the slurry so small that the inlet hose has nothing but atmospheric air to suck in and the pumps "take in" the latter, which necessitates re-priming of the pumping units; (b) a drop of the coefficient of admission of the pumps; (c) non-concurrence of the density of slurries inside the well and on the surface as a result of the air compression.

Complications Arising Due to Temperature and Pressure Changes in the Well

At higher temperatures, there develop in wells conditions which favour an increase in the viscosity of clay muds and cement slurries so that they start thickening more intensively or become solidified.

A temperature fall in wells in the course of flushing must, however, be also taken into consideration, which can bring on complications in casing of wells and, above all, of high-temperature wells. Such sharp temperature fluctuations are seen to occur during flushing of wells after running down casing strings (if no intermediate flushings were practised, or were done but rarely), as well as at the time of pumping cement slurry into the tubing and its egress into the annular space (especially in winter time when cold water is used for mixing of cement). The resulting temperature difference can cause crumbling and even caving-in of the well walls.

When placing cement bridging plugs in deep wells, much consideration must be given to thermal conditions in the well bore: the length of the filling-in pipes diminishes during pumping of low-temperature cement slurries and clay muds. Underestimation of their diminished length results in that during spotting of small cement bridging plugs their top portions are cut off over a great length, the upper perforation holes become exposed and the work done proves futile. In some cases, such carelessness may cause even more serious consequences.

Leaving Large Cement Columns in Wells Following Their Cementing

Because of the use of poor-quality cementing plugs, quite often there develop conditions under which the plugs advance ahead of the cement slurry, this advance being at times quite considerable.

Thus, when use is made of wooden, "collarless" plugs, the most rapidly moving central part of the cement slurry stream entrains the cementing plug, whereas layers adjoining the walls are moving at a lesser speed and lag behind. Consequently, the plug "outruns" the top sections of the cement slurry by tens and even hundreds of metres.

The best way to preclude this type of complications is to use special rubber plugs with which the whole cross section of the casing pipe is filled and they can move on not by gravity, but under some pressure differential.

Complications Arising from Premature Thickening and Setting of Cement Slurry

In the drilling practice this type of complications is of a fairly frequent occurrence.

All other conditions being equal, the thickening of cement slurries is explained by the inconsistency of the chemical and mineralogical composition of cement and improper formulation of the slurry for concrete conditions.

Complications associated with premature thickening and setting of cement slurry have become more frequent in deep wells because of inadequate attention given to their chemical treatment. The process of treatment of the slurry is responsible for some errors made during its preparation, which can become a source of complications. For this reason, serious attention must be paid to the treatment of cement slurries. Their composition must be formulated with the use of an autoclave under conditions that correspond to the ones actually existing in the well.

Many difficulties and failures, particularly in cementing wells with small clearances between the casing and the walls of the well, are in fact due to a certain quantity of water being filtered out, this resulting in a reduced mobility and quicker thickening of the slurry and changes in its other properties.

The viscosity of the intermediate layer (miscibility zone) plays an important role in the rise of pressure during cementing of above all deep, high-temperature wells where both the clay mud and the cement slurry have been treated large amounts of various reagents. Under certain conditions of intermingling, this layer can spread over a sizeable area, while an elevated viscosity will given rise to an excessive pressure during displacement of the plugging mixture.

Long storage of cement slurry tends to retard its setting (when temperature does not exceed 100 °C), increase its viscosity, and possibly create an excessive pressure during its pumping.

Cement-perlite slurries, while mobile under ordinary conditions, change their structural and mechanical properties when subjected to a hydraulic pressure. Already under a pressure of 0.5 MPa, perlite pores are laid bare, water fills them, mobility of the slurry declines, and it becomes thick. It looks as if the slurry undergoes rapid thickening and gets set. But though the setting time of the slurry becomes in this case somewhat shorter, the main disadvantage here is considered to be an increase in viscosity and gel strength. The complications can be forestalled only through a careful formulation of plugging mixtures with due regard for the actual conditions prevailing in the well.

Creating high velocities of the ascending stream of the cement slurry in the annulus leads in a general case to an increase of the pressure on the walls of the well and, as a result of this, the formations undergo hydraulic fracturing. To secure high-quality cementing, in many instances, along with the reduction of the difference between the densities of the cement slurry and the clay mud, it is necessary to lower the speed of the cement slurry movement in the annulus to that of the clay mud during the last flushing of the well. In cementing shallow wells, a decisive factor leading to an upsurge of hydraulic fracturing is the difference in density between the clay mud and the cement slurry. The greater this difference, the more probable the hydraulic fracturing of the formations.

When there are formations in the vertical section of the well susceptible to hydraulic fracturing, an insignificant increase of the hydrodynamic pressure upon the walls of the well results in the loss of the cement slurry.

A greater depth of the wells, diminished gaps between the wall of the well and the casing pipes make it necessary to tackle the problem of fixing the velocity of the ascending stream of the cement slurry in a differential manner, depending upon geological conditions, the design of the well, and the ability of the walls of the well to resist the emerging hydrodynamic pressures.

With a substandard clay mud, losses were recorded in many wells because of its high yield value.

Sharp Pressure Fluctuations in Cementing of Wells

In the course of cementing, pressure sometimes goes up after sudden stoppages of cementing plugs. This type of complications may arise when the inside dimensions of the casing pipes differ significantly. Such complications are most frequent with strings measuring 168 × 127 mm and with the use of ground plugs intented for the 168-mm casing only.

In the process of cementing, there occur sharp drops of pressure. This is due to a variety of causes, *viz.*, squeezing of plugs, stripping of the cement baffle collar, derangement of strings, especially during the displacement of the cement slurry.

The cement baffle collar should sustain the load caused by the impact of the plug being landed. It is also important that the collar and the cementing plug (the metal distance piece) should stand the excess pressure. When using casing float valves, one should carefully watch over the additional filling of the string every 100 to 200 m and it is only after running down the last 200 to 300 m of the pipes that the additional fill may be stopped because of a considerable weight of the landed string.

In the event of a wrongly estimated amount of the displacing fluid, there exists the possibility of a premature seating of the cementing plug onto the cement baffle collar and of a sizeable hydraulic impact followed by the breakdown of the casing. When leaving the cementing head with incompletely turned out set screws, the cementing plug becomes sometimes separated into individual pieces.

15.3. Accident Prevention in Handling Free-Flowing and Dusty Materials

Loading, Unloading and Transportation of Cement

1. The loading and unloading operations, as well as moving of loads should be done under supervision of a staff member responsible for the safety of the operations.

2. Workers with no experience in loading may be admitted to performing loading and unloading operations and to moving weights after having been well familiarized with the pertinent safety regulations.

3. In carrying sacked cement by hand, the maximum weight per worker should not be more than 50 kg for males (80 kg for qualified loaders) and 20 kg for females. Adolescents should not be permitted to engage in loading and unloading of cement. The maximum distance for carrying cement and other materials by hand is not to be more than 50 m on a flat ground.

4. For carrying cement sacks by hand over railroad tracks, a planking not less than 1.5 m wide is to be laid down flush with the rail top.

5. In stacking sacked cement, measures have to be adopted that provide for the stability of stacks and prevent their tumbling down. The permissible height of the stack is 1.5 to 1.7 m (i. e., 10 to 12 rows of standard sacks). The passage between the stacks should not be less than 2m.

6. When loading and unloading operations have to be done at height, the workplaces should be illuminated with flood-lights or other electric luminaires that provide the intensity of illumination of no less than 5 luxes when weights are moved by hand and 10 luxes when the loads are moved with the help of conveyers, trolleys, etc.

7. On completion of operations and, periodically, in the course of protracted loading operations, the floors must be cleared of the cement split from torn sacks.

8. The approach roads to cement sheds are to be arranged in such a manner that the position of the truck body floor and that of the storehouse should be at the same level.

9. Workers engaged in loading and unloading of cement must be supplied with dust-protective garments, aprons made of a thick fabric, and respirators (or gauze bandages).

10. Sacked cement is to be placed in the truck body only if the following requirements are met:

(a) prior to loading, the truck body is to be made clean and should not have any sharp projections (bolts, nails) on the inner surface of its side boards and the floor;

(b) cement sacks should be arranged uniformly over the entire area of the body and not higher than at the level of the truck side boards;

(c) sacks should be placed upright with a slight inclination towards the driver's cab.

11. The sleighs for hauling of cement should be equipped with side boards; the cement sacks loaded on the sleighs must not project above the side-boards level. It is absolutely prohibited for the attendants to sit on top of the loaded sleighs.

12. Besides the driver, only those workers who have received special training are to be admitted to attend to the cement mixers.

13. The site for putting up the cement mixer is to be chosen so as to provide for easy charging of cement and convenient installation of cementing units for pumping the cement slurry. The cement mixer should be placed at a distance of not less than 10 m from current-carrying wires.

14. Before charging a batch of cement weighing more than 10 t, folding jacks available on the mixer should be set in position.

15. When setting up the charging screw conveyer by using a jib crane, one should make sure of the reliability of the hoisting rope.

16. Prior to starting the cement mixer, the dependability of guards on chain transmissions is to be checked.

17. The hopper of the charging screw conveyer is to be put up in such a way as to have its front edge lie flush with the girdle (not above it).

18. Cleaning the conveyer from lumps that find their way into it should be done only with the shut-down engine of the cement mixer; during this operation the driver is to stay outside his cab.

19. In dismantling the charging screw conveyer, the same rules are to be followed as during its assembly.

20. Doing any work at the top of the hopper is permissible only with firmly fixed guard-rails. The movement of the cement mixer with upraised guard-rails is prohibited.

21. It is categorically inadmissible to do any repairs of the moving parts of the cement mixer and to clean its inlet chamber of cement lumps when the machine is in operation.

The safety rules applied in using plugging mixtures are in the main analogous to those cited above. But some features specific for individual materials demand additional preventive measures. Thus, when cement dust precipitating in electric-filter cement kilns is employed as an additive, one should take into account its pungent properties that cause irritation of the mucosa. Therefore, in handling this material dustiness of workplaces is inadmissible.

15.4. Accident Prevention in Cementing Jobs

The cementing of wells is a highly critical operation which takes a relatively short period of time (1 to 3 hours). Therefore, in doing cementing jobs, particular attention is to be paid to safety engineering.

The following are distinctive features of operations associated with the cementing of wells (which determine the establishment of pertinent safety regulations): simultaneous presence of a large number of people; harmful effect of cement and of various chemical agents and aggregates (fillers, especially of organic origin), high pressure under which the process of cementing proceeds, concurrent operation of cementing units and other machines, utilization in certain cases of radioactive substances, the noxious nature of exhaust gases and the noise produced by running engines, as well as work at night (though efforts should be made to do it in the day time only). Cementing at night, even with good illumination, ought to be practised in exceptional cases only.

The cementing units and cement mixers coming out to do cementing jobs should be in good repair. Particular attention is to be given to the proper working condition of their control mechanisms, clutches, and pressure gauges. The use of pressure gauges lacking seals with indication of unexpired inspection date, and with such defects as pointer's deviations and stripped threads should be categorically prohibited.

The diameter of the safety valve pin should be set to definite pressure (commonly 30 % below the maximum pressure under which the pump is designed to operate) at which the work is planned to proceed (provisions being made for a pertinent safety margin). The manufacturing plants should supply sets of pins conformable to the pertinent USSR standard. Each pin must carry a stamp indicating the test pressure. The safety valve must be tested under its specified pressure.

To avert any possible burst of the air cylinder, the cock on the discharge line should be kept open.

To ensure safety of the attending personnel engaged in the operation of cementing unit pumps, the safety valves must be provided with a housing and a discharge pipe for draining the fluid in case of their disruption. Good working order of the safety valve should be checked before departure for the next operation.

The equipment on the platform of the cementing unit should be properly placed and firmly fastened. It must not stand in the way of the driver and the fitter during their work.

With the operator attending the cement mixer, side guards should be set up and fixed in place and the discharge holes (with a diameter of 495 mm) should be covered with a special 20 × 20 mm mesh grate.

The spare wheels of the machine should be put up and fixed on brackets; their taking off or putting up on the brackets is to be done only by means of winches of the cementing or mixing units.

The equipment placed on the platform of the cementing units with revolving assemblies is to be covered. The piston and vertical pumps, drives and the upper engine are to be covered with lightweight shields. A stage for the worker keeping watch over the level of the fluid in the receiving tank is to be arranged at the measuring tanks (between the pump and the tank).

Particular attention should be paid to arranging an enclosure when operating cement mixers Model CMH-10; they have no protective guards of their own, since they are not intended for being loaded at the drill rig. It is advisable to use special collapsible or straight ladders when operating both the cementing units and the cement mixers, for their platforms lie high up above ground level. These ladders can be used only at the time when the cementing units and the cement mixers are parked. Particular attention should be paid to the proper condition of steps, guard rails, and holders when the machines are moved about at the parking place and in the course of cementing jobs.

One of the indispensable appliances of the CMH-20 Model cement mixer is a special screw conveyer. In the traveling position it must be fixed with special plates in the rack. It should not be fastened with cords or wire.

The discharge and delivery pipes 2 to 4 m long are transported together with the cementing units and are part of the cementing unit equipment. They are kept in slotted recesses and are to be clamped with bolted planks or with latches.

When filling the cementing unit tanks with water and the cement mixers with plugging cement, jacks are to be set in position, all the safety regulations being adhered to.

When putting up the cementing units and cement mixers on the ground, this is to be cleared of all extraneous objects.

The units and mixers should not be placed with their cabs facing the well.

The distance between the drill rig and the cementing units should be kept to a minimum.

To avoid vibration of the units during operation of the pump, timber bars are to be placed under the wheels of the machines.

To facilitate the work of the attending operators of the cement mixers, the latter should be set up in such a way as to make their back wheels approximately coincide with the axle of the front wheels of the cementing units operating in pair with them.

It is prohibited to stand underneath the charging device when this is being lifted.

Prior to starting the charging screw conveyer, the presence and the conditions of enclosures on all external moving parts are to be inspected.

It is prohibited to adjust the screw conveyer and clean it during the operation of the engine and transmissions.

The mixer should not be moved when loaded with more than 12 t of cement.

The connections of the cementing units are allowed to be made only after the string has been run down the well. No one is to be permitted to stand on the access boards of the drill rig when running down the string and restoring circulation.

The connections of the cementing unit with the wellhead should be "soft", i. e., flexible bends must ensure absorption of hydraulic impacts in the line during operation of the pumps. No crossing of discharge lines is to be admitted.

When tying discharge lines by quick-detachable connections, no one is allowed to be in front of operating hammers. Water supplied to the mixing arrangement should be fed via the equalizer only through rubber hoses.

At night, the workplaces should be lighted by lamps with illuminating intensity of not less than 5 lx. All the operations that involve laying down and assembly of pipelines, mounting of the screw conveyer, ladders, etc. are to be performed by men wearing gauntlets and protective garments.

Testing of pipelines under pressure begins by the word of command of the person in charge of the operations after all members of the attending crew have left the ground to a safe distance. Concurrent elimination of gaps in the line and its testing are prohibited. After their testing under pressure, the pipes of discharge lines must be rigidly fixed to the legs of the derrick so as to preclude their swinging.

The towing of the cementing units and cement mixers is permitted to be done only with a rigid tow line no less than 2.5 m long tied to the central hook at the bumper of the machine. Coupling and uncoupling of the transport facilities on the move are prohibited.

15.5. Safety in Handling Radioactive Isotopes

During its preparation and pumping into the well, the activated slurry should on no account be allowed to get on the body and garments of the operators; the slurry should not be split over the territory of the drill rig.

The ampoules with the radioactive isotope are to be crushed with special nippers having long handles only upon their immersion into a tank filled with water to a depth of 20 to 30 cm below its level.

The addition of the radioactive fluid into the tank of the cementing unit is to be allowed only after the discharge lines have been tested under pressure and the pump has been primed.

On termination of the work, the tank, water and cementing pumps, and the rest of the equipment, along with the hydraulic mixing arrangement and the discharge pipe of the cement mixer, should be thoroughly washed with pure water. Thereupon, the equipment, the territory around the well, and the garments of the operators must be examined for the presence of any dangerous radioactive contaminants.

The remnants of unused or split radioactive solutions (fluids), as well as the residue left over after washing of the units, ought to be diluted with water down to permissible concentration and removed beyond the boundaries of the drill rig.

15.6. Safety Regulations to be Observed When Working in Gaseous Environment and Handling Chemicals

With the cementing unit running at the drill rig, smoking and the use of open fire are categorically prohibited. Exhaust pipes of the units' engines should be provided with spark arresters.

Prior to handling dry salts and their highly concentrated solutions (10 to 20 %), an ointment made up of a mixture of paraffin, lanoline, and phenol is to be applied to the skin of the hands. Should such an ointment be lacking, the wearing of rubber gloves is obligatory.

When preparing a solution of a dry chromate, the workers should invariably make use of respirators of the Ф-45 or Ф-46 types.

On completion of the work, the hands should be washed with a 5- % sodium hyposulphite solution or a 10- % sodium sulphite or bisulphite solution. Persons dealing continually with chromates and bichromates should be made subject to an examination by an E. N. T. physician every month and to a general medical examination once in 6 months.

Persons suffering from affections of the nasal cavity, chronic laryngitis, tuberculosis, chronic bronchitis, bronchial asthma, and from any form of eczema should not be permitted to handle dry chromates and bichromates.

15.7. Safety Regulations to be Observed when Working in Winter Time

1. To protect the hands against frost-biting, all the operations involving contacts with metals have to be done in gauntlets.

2. At the end of steam pipelines (hoses) a small metal tip with a valve should be provided. Once warming of the pipeline has been completed, the valve should be shut so as to avert scalding of the workers with steam. It is prohibited to leave the hose open with steam issuing from it, i. e., with an open valve.

3. The passage of steam heats the metal nozzle at the end of the hose. The worker engaged in warming up of the equipment should hold the steam hose by its rubber part and not by the metal tip.

4. The pressure in the steam boiler is to be maintained at a level corresponding the quantity of steam consumed.

5. The machine components are to be warmed up with steam uniformly from all sides.

6. To avert scalding of the face with steam or with sprays of hot water, no outsiders should be permitted near the site where machine components are being warmed up.

7. When starting engines by hauling the units, all the rules for hooking the towing line and towing of the machines should be observed.

8. The end of the steam hose let down into the measuring tank for heating of water must be firmly fastened to prevent its jumping out.

9. To preclude freezing of the pumps, an all-round circulation of the fluid (heated water from the measuring tanks) has to be maintained with the pump drives running at a low speed until cementing is started.

10. In the course of cementing, the cementing head and the pressure gauge at the head should be warmed up periodically. Prior to squeezing the cementing plug through the top valve. the head is to be thoroughly steamed.

15.8. General Safety Rules

1. All persons not engaged in the cementing work should be kept away, beyond the boundaries of the drill rig and outside the area of the units' action and their distribution (supply) lines.

2. The transport of workers on the upper platform of cementing units or on the roof of cement mixers is categorically prohibited.

3. The crew must have at their disposal a medicinal chest, in addition to the one available for the driller's team. The medicinal chest is to be kept in the cab of one of the units.

4. All the crew members of cementing units and cement mixers must pass examination on matters concerned with safety engineering. With the arrival of a cementing unit or a cement mixer of new design, all the crew members must be given an additional briefing in the scope sufficient for doing work on their own.

5. When doing cementing jobs in a complicated well, or with new elements introduced in the operations, the workers should be given an additional briefing in which the essence of the new elements and of preventive safety measures should be explained.

Chapter 16

Cementing Quality Check

Cementing quality check, also known as cement bond logging, is the final operation in cementing wells. As the depth of wells increases and their cost grows higher, the cement bond logging methods are constantly gaining in importance. The indispensable condition here is the rapidity of checking the qauality of cementing jobs.

The absence of water influx and the leak tightness of the casing string in themselves are not testimony to the success of cementing. The quality of cementing jobs depends on many factors. The most general characteristics indicative of the quality of cementing (where the casing string is leak-tight) are the extent of displacement of the drilling mud by the cement slurry in the annular space of the well, the density (impermeability) of cement stone, and its durability in the well annulus.

The methods for checking the quality of cementing the annular space of the well prior to its stimulation (and also after recementing jobs) are temperature logging, acoustic and radioactive (nuclear) cement bond logging, casing leakage tests by internal or external pressure (these tests are also conducted after remedial cementing jobs), cement bridging plug tests by load from tubing (or drill pipes) landed on them, both with and without concurrent flushing.

1. Temperature logging is one of the first methods for checking the annular cement top (level), or fill-up height. The method is based on recording temperature abnormalities in the well opposite to the cement mass in the annulus, caused by the liberation of heat in the course of its hardening. Therefore, temperature logging should be carried out during the period of hardening of cement slurry. This time is different for different slurries and should be determined beforehand under laboratory conditions. Temperature measurements are taken by means of a downhole electrical thermometer run into the well on a logging cable.

To improve the accuracy of checking for the presence of cement stone in the annular space, the temperature log, recorded while waiting on cement, should be checked against a caliper log or a section gauge log. In cemented hole sections, the configuration of the temperature log is in line with that of the caliper or section gauge log, whereas in uncemented hole sections, these logs are at variance.

Limitations of the method: impossibility of checking the annular cement level after hardening of the cement slurry and low effectiveness in cases where cementing is done with lightened plugging mixtures that evolve inadequate quantities of heat during hardening and also in high-temperature wells. Moreover, while waiting on cement, it is difficult to obtain information, in addition to that on the annular cement level, about the character of cement mass distribution in the annular space on the basis of a single-run temperature logging data.

2. Radioactive (nuclear) cement bond logging is based on the properties of radioactive substances, and its methods may be divided into the following two groups.

(a) Activation (radioactive tracer) logging methods whereby the cement slurry is activated with some radioactive isotope (usually a short-lived one) and then the character of its distribution in the annular space is investigated by measuring the radiation intensity with the aid of gamma-ray logging.

These methods are usually used where the quantities of cement slurry required are not very large (stepwise cementing, remedy-isolation works, etc.).

An improved version of such methods is one involving the use of a gamma-ray cement log bond sonde, developed in this country, for a detailed investigation of the character of distribution of activated cement slurry in the annular space of the well. This instrument helps evaluate changes in the thickness of the cement annulus along the periphery of the casing string at any point of the well by measuring the intensity of collimated gamma radiation with the aid of a scanning device.

The disadvantage of activation logging methods is the high radiation level remaining for a comparatively long period of time, which prevents any other radiometric surveys being conducted in the well. The radioactive tracer logging has not found wide application because radioisotopes are difficult to handle and present radiation hazard.

(b) Gamma-gamma-ray cement bond logging methods depend upon the relation between the intensity of scattered gamma radiation and the density of the scattering medium. Gamma-gamma-ray cement bond log sondes rely for their operation on the fact that some of the gamma radiation issued by their radiation sources is picked up by their detectors after its being scattered in the drilling mud, cement slurry, casing string, and, sometimes, rocks. If cement slurry or stone is present in the annular space, the intensity of the radiation picked up by the detectors will be lower than in the case of drilling mud. These methods are widely used in this country and enable one to determine, in addition to the annular cement level, the character of distribution of cement slurry in the annular space (the presense of channels in cement) and also the eccentricity of the casing string in the well bore.

In wells cased with pipes 146 or 148 mm in diameter, investigations are carried out with the aid of the ЦМТУ-1 Model cement bond log sonde.The downhole instrument includes a pickup assembly of three gas-discharge counters equally spaced (at 120°) around the periphery of a lead shield, which makes it possible to record changes in the intensity of scattered radiation along three generatrices of the well simultaneously.

The ЦММ-3-4 Model cement bond log sonde of similar construction is intended for use in casing strings 89 to 114 mm in diameter.

The ЦФ-4 Model cement bond log sonde, intended for investigations in large-diameter casing strings, has four shoes that are pressed against the casing walls when taking measurements. Housed in special recesses in the shoes are four pickups. The presense of the fourth measuring channel is the only difference between the ЦФ-4 and ЦМТУ-1 Model instruments.

A more effective cement bond log sonde, called cement flaw detector, has recently become available in this country. The instrument is equipped with collimation emitters and detectors rotating in synchronizm. The use of such a scanning device provides for a maximum differentiation of the recorded log with respect to the density of the material in the annular space and reduces the effect of distorting factors.

However, the above instruments are sensitive to changes in the casing wall thickness. Thus, a 1-mm change in the casing wall thickness alters cement bond log sonde readings by as much as 10 to 20 %, which makes it necessary to measure additionally the casing wall thickness.

In this connection, a two-channel combination downhole gamma-gamma-ray cement flaw detector-casing wall thickness gauge, Model СГДТ-2, has been developed. The gamma-gamma-ray casing wall thickness gauge operates on the same principle as the cement flaw detector, but uses a softer radiation and has a smaller distance between the emitter and detector whose collimation windows are arranged at an angle to the instrument axis.

The combination in the СГДТ-2 Model instrument of a collimation scanning device and a casing wall thickness gauge enables one to determine, under favourable geological and technical conditions, the density of the material in the annular space of the well accurate to within 0.1 to 0.2 g/cm^3 and detect channels in cement stone whose cross-sectional area is only 2 % of that of the annular space. In contrast, the ЦМТУ-1 Model cement bond log sonde is capable of detecting only channels whose cross-sectional area is no less than 10 % of the cross-sectional area of the annular space.

Factors limiting the application of gamma-gamma-ray cement bond logging are as follows:

(a) for the gamma-gamma-ray instruments to operate properly, there should be a considerable difference both in density (0.3 to 0.5 g/cm^3) between the cement stone and the drilling mud and in diameter (4 to 5 cm) between the well bore and the casing string;

(b) scintillation counters are capable of operating reliably only at temperatures of 80 to 100 °C, therefore the use of the gamma-gamma-ray instruments (especially of the СГДТ-2 Model) is restricted mainly to relatively shallow wells.

In the USA, the gamma-gamma-ray cement bond logging is effected mainly by means of single-channel instruments that record the total intensity of scattered radiation around the periphery of the well and thus have a lower resolving power than the instruments of the Soviet make.

3. Acoustic cement bond logging is at present the most effective method for evaluating the quality of cementing wells, for it makes it possible to determine not only the boundary between the cement stone and the drilling mud, but also the condition of the contact between the cement stone and the casing string and, sometimes, also between the cement stone and the surrounding rocks.

The acoustic cement bond logging depends on the relation between some characteristics (velocity, amplitude, and frequency) of acoustic vibrations, which are being emitted by the radiator of the cement bond log sonde and picked up by its receiver, and the rigidity of the bond between the casing string and the rocks and the elasticity of the medium in the annular space of the well.

Most widely used in this country are acoustic cement bond log sondes Model АКЦ-1, designed for pressures up to 60 MPa and temperatures of 120 to 150 °C, and Model АКЦ-2, designed for pressures up to 100 MPa and temperatures of 150 to 200 °C, that are run into the well on a three-conductor logging cable.

Acoustic cement bond log sondes for deep wells, Model АКЦ-4 (for pressures up to 1200 MPa) and Model АКЦ-60 (for wells up to 7000 m in depth) are now being developed and prepared for serial production. These instruments are designed for use in conjunction with a single-conductor logging cable.

By taking repeated measurements with an acoustic cement bond log sonde, one can investigate the course of cement stone formation behind the casing string in time and study also the effect of mechanical actions that the casing string is subjected to (perforations, pressure, fluctuations in the string, etc.) on the quality of well cementing.

However, acoustic cement bond log sondes have a number of inherent disadvantages that make it generally difficult to evaluate unambiguously the quality of cementing on the basis of their data. The principal shortcomings of these instruments are as follows:

(a) inadequate informational value of the recorded parameters, which makes it difficult both to assess the condition of contact between the cement stone and the surrounding rocks and to take account of the distorting effect of the measurement conditions in the well and instrumentation factors;

(b) inadequate resolving power of the instruments, which makes them incapable of detecting defects (channels) in the cement stone that are less than 40° in opening angle relative to the well axis.

To raise the informational value of the acoustic cement bond logging data, both in the USSR and other countries (USA) there have been developed special attachments to the surface instrumentation (e. g., the AKP Model cine recorder developed in this country) that make it possible to record the total acoustic signal picked up by the receiver of the cement bond log sonde. By interpreting the characteristics of such a signal, one can assess with a fair degree of assurance the condition of contact between the cement stone and the surrounding rocks and take into account the effect of distorting factors on the measurement results.

Also developed in this country is a combination photorecorder which simultaneously records on a single logging film a cement bond log and a phase correlogram indicative of the changes that the total acoustic signal suffers with increasing depth of the well.

To improve the resolving power of the acoustic cement bond logging method, a scanning device for an acoustic cement bond log sonde has been developed in the USSR, which enables one to investigate changes in the contact between the cement stone and the casing string around its periphery and detect defects in the stone that an ordinary cement bond sonde cannot discern.

To detect channels in the cement stone behind the casing string, there has been developed in the USA a combination acoustic cement bond log-neutron log sonde. An increase in the porosity of the cement stone in the annular space, detected on the basis of neutron logging data, is an additional indication of a channel.

Combining different methods is one of the most promising ways to further improve the effectiveness of both the cementing quality check and the overall control over the quality of well casing and cementing.

4. Casing string leakage tests. Cemented casing strings are tested for both leak tightness and strength by subjecting them to an excessive pressure, either internal or external, which in the field practice is effected either by pumping a liquid or gas into the string being tested or by drawing down the fluid level in the string.

Surface casing and intermediate strings are usually tested for leakage by building up an excessive hydrostatic pressure in them, whereas production (flow) strings are tested by both methods, i. e., by creating an excessive internal pressure and by drawing down the fluid level in them. The second method of testing production strings is used in addition to the first one, in cases where no excessive pressure is anticipated to develop at the wellhead during testing and operation, and also in all exploratory wells.

The magnitude of the excessive pressure in leakage testing of casing strings must be 20 % in excess of the internal string pressure adopted in designing a particular string for strength. In some cases, the leakage test pressure for surface casing and intermediate strings carrying blowout preventer equipment is specified proceeding from the maximum expected wellhead pressure that may occur in the process of development and elimination of surface gas and oil shows and blowouts and also in the course of other operations carried out in the well. The magnitude of this pressure is specified individually for each deposit, with due regard for the scope of the geological exploration and technical survey work that has been done in the region, characteristics of the blowout preventer equipment used, and other pertinent factors. But in all cases, the magnitude of the excessive internal pressure in testing casing strings for leak tightness must be no lower than indicated below.

Casing OD, mm	377-426	273-351	219-245	178-194	168	141-146	114-127
Excessive internal pressure, no less than, MPa	50	60	70	75	90	100	120

Surface casing and intermediate strings are leakage tested prior to drilling out the cement plugs left at their bottom. To test the cemented annulus (cement sleeve or column) for leakage, the string is tested once more after the cement plug has been drilled out and the well sunk 1 to 2 m below the casing shoe.

To prevent hydraulic fracturing of formations in the vicinity of the casing shoe, the magnitude of the internal string pressure in the repeated test is determined from the relation

$$p_{test} = p_{cs} - 0.1\rho H$$

where p_{test} is the lest pressure at the wellhead, MPa, p_{cs} is the maximum expected internal pressure at the casing shoe in the event of a blowout or flowing of the well, MPa, ρ is the density of the fluid in the casing string, g/cm^3, and H is the landing depth of the casing shoe, m.

Should the calculated magnitude of the test pressure prove to be negative or should the casing shoe be located, for technical reasons, in a previous rock zone, the repeated leakage test is not conducted. In this case, the possibility of sinking the well further down and drilling in formations presenting blowout hazard is determined by the executive staff of the drilling enterprise. If the test fluid is lost into the formation in the course of the repeated leakage test, a remedial-isolation work is done in the casing shoe interval of the well.

Production casing is tested for leakage after determining the location of the top of the cement plug, drilling the plug out to the desired depth (if necessary), and replacing the drilling mud with water.

In carrying out an additional draw-down test of production casing, the minimum depression of the fluid level in the string should be as indicated below.

Well depth, m	500	500-1000	1000-1500	1500-2000	2000
Minimum depression of fluid level, m	400	500	650	800	1000

In wells drilled with the use of a drilling mud having a density of 1.4 g/cm^3 and more, it is permissible not to subject the production casing to the draw-down test, but simply replace the mud with water and then monitor the position of its level at the wellhead.

When testing casing strings for leakage by internal pressure, a string is considered leak-proof if the test pressure built up in it remains unchanged for 30 minutes, or if it drops during this time by no more than 0.5 MPa for test pressures in excess of 7 MPa or by no more than 0.3 MPa for test pressures below 7 MPa. The monitoring of the pressure in the string should be started 5 minutes after its magnitude has been brought up to the specified test value.

In the draw-down test, a casing string is considered leak-proof if the rise of the fluid level in it after the expiration of an 8-hour observation period does not exceed the values indicated in Table 37.

Table 37

Maximum Permissible Rise (in Meters) of Fluid Level in Casing Strings During Draw-Down Test

Casing OD, mm	Fluid level depression in test, m				
	400	400-600	600-800	800-1000	1000
114-219	0.8	1.1	1.4	1.7	2.0
219	0.5	0.8	1.1	1.3	1.5

To exclude the effect of the water running down the casing walls, the monitoring of the fluid level in the casing should be started 3 hours after it has been drawn down. When testing, the fluid level in the casing should be measured every 2 hours.

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